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Prebond Inspection Techniques to Improve the Quality of Adhesive Bonding Surface Treatments

Andrew N. Rider

**Air Vehicles Division
Defence Science and Technology Organisation**

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ABSTRACT

Adhesively bonded repairs to metallic aircraft structure can be used in a variety of applications to solve difficult maintenance issues where traditional mechanically fastened repairs are often unsatisfactory. For example bonded repairs have been applied to reduce stress intensity in fatigue prone areas of aircraft and, thereby, extend service life of the component, providing substantial maintenance savings. Despite their valuable contribution to aircraft maintenance, bonded repairs are treated as fail-safe items when used on primary aircraft structure. One of the reasons for the lack of credit for bonded repairs is the absence of a reliable non-destructive inspection (NDI) technique that can guarantee bond quality and strength. One solution to reduce this problem is the development of objective prebond inspection techniques that can guarantee the quality and reliability of the critical surface treatment process applied prior to the adhesive bonding operation. The use of a gloss-meter unit and surface quality monitor to quantify the prebond condition of metallic substrates is an effort to further improve the reliability and reproducibility of current bonding operations.

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Prebond Inspection Techniques to Improve the Quality of Adhesive Bonding Surface Treatments

Executive Summary

Adhesively bonded repairs to metallic aircraft structure can be used in a variety of applications to solve difficult maintenance issues where traditional mechanically fastened repairs are often unsatisfactory. For example, bonded repairs have been applied to reduce stress intensity in fatigue prone areas of aircraft and, thereby, have extended the service life of the component, providing substantial maintenance savings. Despite their valuable contribution to aircraft maintenance, bonded repairs are treated as fail-safe items when used on primary aircraft structure. One of the reasons for the lack of credit for bonded repairs is the absence of a reliable non-destructive inspection (NDI) technique that can guarantee bond quality and strength. One solution to assist in reducing this problem is the development of objective prebond inspection techniques that can guarantee the quality and reliability of the critical surface treatment process applied prior to the adhesive bonding operation.

In this report a commercially available gloss-meter unit and surface quality monitor have been employed to quantify the prebond condition of metallic substrates. The ability to objectively quantify the surface condition of the metal substrate prior to the adhesive bonding step is critical to guaranteeing the reliability and reproducibility of current bonding operations. The development of a database that can then be used to relate the measured bond performance to the quantifiable measurements of the surface can then be used to reduce the risk of producing inferior quality bonded repairs in the field. The gloss-meter unit can be employed to measure the quality of the abrasion and grit-blasting steps, which provide the critical mechanical roughening of the surface prior to bonding. The surface quality meter can be used to measure the cleanliness of the surface before and after treatment with the organosilane coupling agent. The coupling agent is used to improve the hydrolytic resistance of adhesive bonds formed between metal and epoxy adhesive, the adhesive systems typically used in bonded repairs.

The following report provides a summary of the gloss and surface quality meter measurements of clad Al2024-T3 aluminium alloy during the initial grit-blasting and abrasion steps and the chemical activation step resulting from organosilane application. The purpose of the measurements was to establish the range of values that would occur when a trained technician was attempting to perform the pretreatment steps to current standards and to relate these ranges to bond quality. Bond quality was assessed with the wedge test and is currently used by the Royal Australian Air Force (RAAF) in the training and requalification of technicians involved in Australian Defence Force (ADF) bonding activities.

Results of gloss-meter measurements on abraded and grit-blasted surfaces revealed that the gloss readings were sensitive to the level of abrasion and grit-blasting, with measurements ranging from over 500 gloss units for an "as-received" solvent cleaned aluminium surface to between 2-3 gloss units for a satisfactorily grit-blasted surface. Additional experiments carried out with a scanning rig that had displacement control in the X, Y and Z directions revealed that abrasion and grit-blasting were relatively homogeneous over a 6" by 6" square plate. However, some specific measurement geometries were very sensitive to the height of the gloss-meter unit above the sample. The signal dropped significantly for light reflected at a grazing incidence when the measurement height was greater than 0.3 mm.

Measurements of the surface cleanliness of the aluminium surface after abrasion, grit-blasting and treatment with the organosilane coupling agent was undertaken with the surface contaminant meter (SQM-200). Results indicated that the signal was very sensitive to the environmental exposure time after abrasion or grit-blasting. Typically, the signal would drop at a logarithmic rate. Measurements to assess the homogeneity of the surface cleanliness were performed with a specially designed X-Y scanning unit and associated control software. Maps of signal versus position on the 6" by 6" aluminium plate indicated that the surface treatment provided a uniform cleanliness. Wedge test results for plates that had been treated and measured using the gloss-meter and surface quality meter indicated that the current procedure is capable of providing repeatable bond quality and durability.

It is proposed that the gloss-meter readings be phased into bonding operations using the RAAF grit-blast and silane treatment. Initially, gloss measurements of grit-blasted wedge test plates produced during training and requalification testing of ADF bonding technicians could be implemented. Eventually, it is anticipated the gloss meter and surface contaminant meter could be incorporated into processes used for undertaking bonded repairs on primary aircraft structure in order to guarantee the reliability of adhesive bonded repairs in service.

Authors

Andrew Rider

Air Vehicles Division

Andrew Rider joined DSTO in 1988. He has a PhD in Physical Chemistry from the University of New South Wales and is currently employed as a Senior Research Scientist with Air Vehicles Division. He is involved in research to support RAAF maintenance of bonded structure and application of composite repairs used on ADF aircraft.

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1. Introduction

Adhesively bonded repairs to metallic aircraft structure can be used in a variety of applications to solve difficult maintenance issues where traditional mechanically fastened repairs are often unsatisfactory. For example, bonded repairs have been applied to reduce stress intensity in fatigue prone areas of aircraft and, thereby, have extended the service life of the component, providing substantial maintenance savings [1]. Despite their valuable contribution to aircraft maintenance, bonded repairs are treated as fail-safe items when used on primary aircraft structure. One of the reasons for the lack of credit for bonded repairs is the absence of a reliable non-destructive inspection (NDI) technique that can guarantee bond quality and strength [2]. One solution to assist in reducing this problem is the development of objective prebond inspection techniques that can guarantee the quality and reliability of the critical surface treatment process applied prior to the adhesive bonding operation [3].

In the following report a commercially available gloss-meter unit and surface quality monitor were employed to quantify the prebond condition of metallic substrates. The ability to objectively quantify the surface condition of the metal substrate prior to the adhesive bonding step is critical to guaranteeing the reliability and reproducibility of current bonding operations. The development of a database that can then be used to relate the measured bond performance to the quantifiable measurements of the surface can then be used to reduce the risk of producing inferior quality bonded repairs in the field. The gloss-meter unit could be employed to measure the quality of the abrasion and grit-blasting steps, which provide the critical mechanical roughening of the surface prior to bonding. The surface quality meter can be used to measure the cleanliness of the surface before and after treatment with the organosilane coupling agent. The coupling agent is used to improve the hydrolytic resistance of adhesive bonds formed between metal and epoxy adhesives.

The following report provides a summary of the gloss and surface quality meter measurements of clad Al2024-T3 aluminium alloy during the grit-blasting, abrasion and organosilane application steps. The purpose of the measurements was to establish the range of values that would occur when a trained technician was attempting to perform the pretreatment steps to current standards and to relate these ranges to bond quality. Bond quality was assessed with the wedge test and is currently used by the Royal Australian Air Force (RAAF) in the training and requalification of technicians involved in Australian Defence Force (ADF) bonding activities [4].

2. Experimental

2.1 Gloss-meter measurements

2.1.1 Reference plate measurements

A previous report identified a gloss-meter unit that was well suited to discriminating the different levels of abrasion and grit-blasting on aluminium surfaces [5]. The hand held Micro TRI Gloss® gloss-meter unit (Figure 1) was purchased from BYK Gardner [6]. The unit can take gloss measurements at three angles relative to the surface normal (20°, 65° and 80°), is self-calibrating and can download measurements to a spreadsheet application. Measurements comply with ISO, DIN and ASTM standards.



Figure 1 *Micro TRI Gloss gloss-meter unit from BYK Gardner, used to measure aluminium surface abrasion and grit-blast quality.*

The basic principle of operation of gloss-meters relies on the measurement of the total amount of specularly reflected light from a surface. The gloss-meters measure the quantity of reflected light at different angles, typically 20, 60 and 85 degrees. The measurement angle is used in inverse proportion to the level of surface gloss, with glossy and matt surfaces being better discriminated at 20 and 85 degrees, respectively. As the surface gloss decreases, the level of dispersed reflection increases. The relationship between incident and reflected light is complex (equation 1):

$$L_{\lambda}(\hat{\theta}_r) = \int R_{\lambda}(\hat{\theta}_i, \hat{\theta}_r) L_{\lambda}(\hat{\theta}_i) \cos \theta_i d\omega_i \quad (1)$$

In equation 1 $L_\lambda(\hat{\theta}_i)$ and $L_\lambda(\hat{\theta}_r)$ represent the local incident and reflected light with angle $\hat{\theta}_i = (\theta_i, \phi_i)$ and $\hat{\theta}_r = (\theta_r, \phi_r)$. λ is the wavelength of light, $d\omega_i$ is the solid angle of incident light and R_λ is the bidirectional reflection distribution function [7]. From equation 1 it can be seen that perceived gloss is affected by wavelength as well as the angles of incidence and reflection of the light. In practice, gloss meters work on the basis of defined reflection angles and the use of a polished black glass calibration standard. The polished black glass provides a value of 100 gloss units, which is typical of a high gloss non-metallic paint and the gloss units are define by equation 2:

$$Gloss \ units = 100 \frac{\Phi_{\theta, R_\lambda^{Sample}}}{\Phi_{\theta, R_\lambda^{Reference}}} \quad (2)$$

Where $\Phi_{\theta, R_\lambda^{Sample}}$ and $\Phi_{\theta, R_\lambda^{Reference}}$ are the reflection influx of the sample and the reference surface through the photodetector aperture.

Twelve plates were treated with combinations of methyl ethyl ketone solvent (MEK) cleaning, abrasion using a Scotchbrite® abrasion pad and grit-blasting at 50 psi using 50µm alumina grit from a distance of 10 to 15 cm. The gloss measurements were performed on an X, Y, Z scanning rig at 30 positions across a 6" by 6" clad Al2024-T3 aluminium plate at three heights of 0.35, 0.65 and 1.0mm. The gloss-meter is designed to operate in contact mode, however, for use in surface treatment applications contact of the gloss-meter with the surface could provide a source of unwanted contamination. As a result of these concerns, studies examined the potential of the gloss-meter unit to be used in non-contact mode. The variation in gloss-meter readings as the stand-off height was adjusted was examined to determine if a simple adjustment to the gloss-meter base could be made. For example, four small sapphire beads of a prescribed height could be attached to the gloss meter base to minimise surface contamination without compromising the measurement accuracy..

The combination of treatments and measurements are shown in Table 1. Measurements were performed parallel and normal with respect to the rolling direction of the 3 mm thick aluminium plate.

Table 1 Surface treatments applied to clad Al2024-T3 aluminium alloy that were measured using the Micro TRI Gloss® gloss-meter unit

Treatment No.	Solvent Clean	Abrasion direction P/N-parallel/normal to rolling direction (RD)	Grit-blast P/N-parallel/normal to rolling direction (RD)
1.	MEK	---	---
2.	MEK	---	Grit-blast, P
3.	MEK	Light abrasion, P	---
4.	MEK	Medium abrasion, P	---
5.	MEK	Medium abrasion, P	---
6.	MEK	Medium abrasion, N	---
7.	MEK	Heavy abrasion, N	---
8.	MEK	Light abrasion, P	Grit-blast, P
9.	MEK	Medium abrasion, P	Grit-blast, P
10.	MEK	Medium abrasion, P	Grit-blast, P
11.	MEK	Medium abrasion, N	Grit-blast, N
12.	MEK	Heavy abrasion, N	Grit-blast, N

2.1.2 Measurement of BSTT wedge test plates

In addition to the measurements taken on the plates listed in Table 1, plates manufactured during requalification and testing of bonding technicians by the Bonded Structures and Testing Team (BSTT) were also examined. As a part of requalification and testing each technician manufactured a wedge test. A strip of flash breaker tape was placed at the bottom of each wedge panel after grit-blasting, but prior to bonding. The tape enabled one side of each panel to be measured with the gloss-meter after testing and the process is detailed in Figure 2. The measurement of the grit-blasted surface with the gloss-meter then provided a direct link between grit-blast quality and bond quality, as measured by the wedge test.

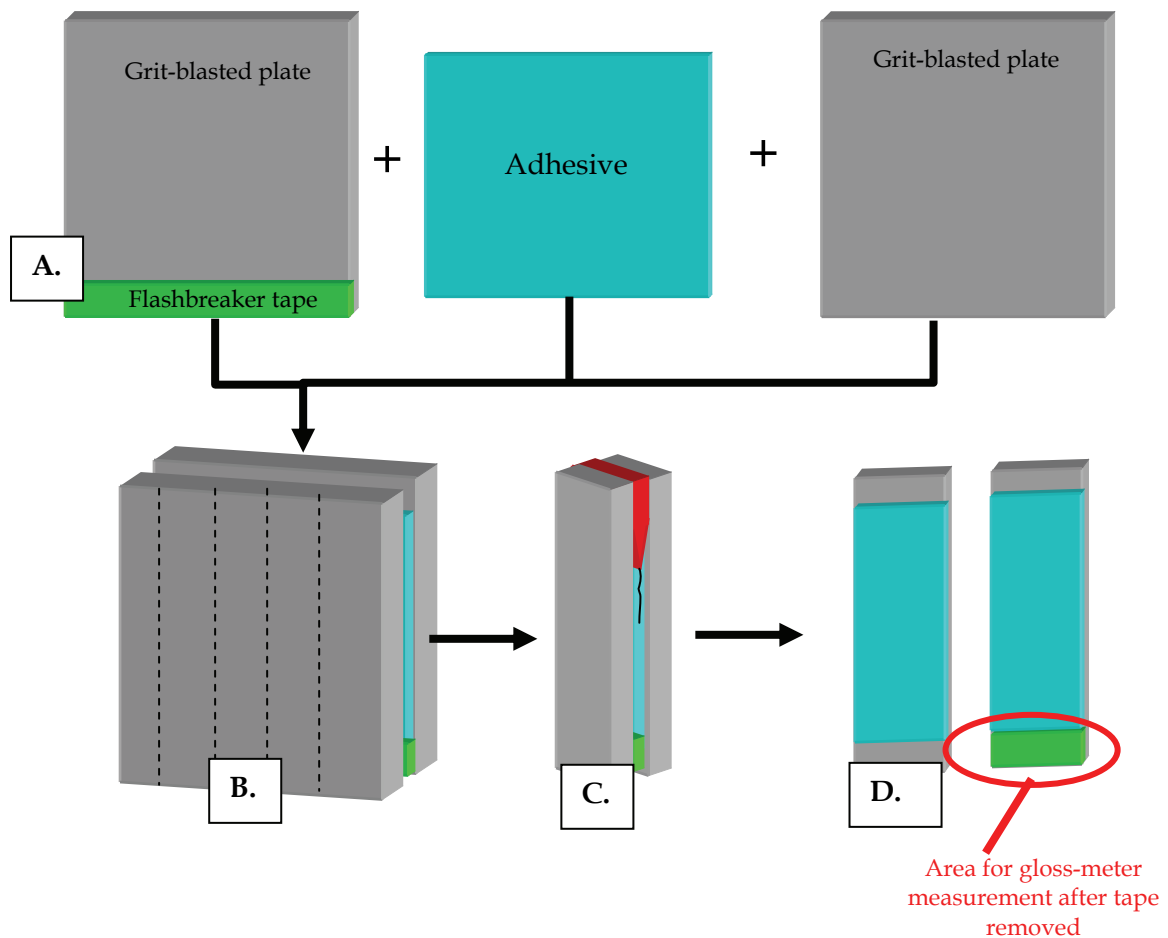


Figure 2 Process of gloss-meter measurement of grit-blast quality on wedge test plates manufactured during BSTT requalification and testing. (A) Flashbreaker tape placed on 1 plate prior to bonding protects the grit-blasted surface during (B) bonding, specimen cutting, (C) testing and is (D) removed after individual fingers are separated.

2.1.3 Measurement of plates before bonding

As a part of previous work that has examined optimising the current grit-blast and silane surface treatment used by the RAAF for adhesive bonded repairs to metallic structure [8], a series of wedge tests were manufactured in which several important parameters were adjusted. During the fabrication of the wedge test plates that examined the variation of these manufacturing parameters, a series of gloss-meter measurements were undertaken during the grit-blasting stage of the surface treatment procedure. This provided a direct correlation between grit-blast quality and bond quality, as measured by the wedge test. Details of the grit-blast and silane surface treatment procedure used by the RAAF are provided in Appendix A: [4].

2.2 Surface Quality Measurements

The surface cleanliness of the aluminium plates prepared for bonding using the grit-blast and silane procedure (Appendix A:) was examined using the SQM-200 from Photo Emission Technologies [9]. Figure 3 shows the principle of operation for the SQM-200, which is based on the photoelectric effect, equation 3, where E is the photoelectron energy, \hbar is Planck's constant and ϕ is the work function of the surface.

$$E = \hbar\nu - \phi \quad (1)$$

During operation, low energy UV photons are directed at the surface of interest from a mercury vapour lamp with two emission peaks at approximately 5.0 and 6.7eV. Photoelectrons are emitted with a kinetic energy equal to the incident photon energy minus the work function of the surface of the material being examined. In principle materials with a work function lower than approximately 7 eV can be examined with the SQM-200. Electrons emitted from the surface are attracted towards the collector biased at approximately 40V and the current is measured with a solid state electrometer.

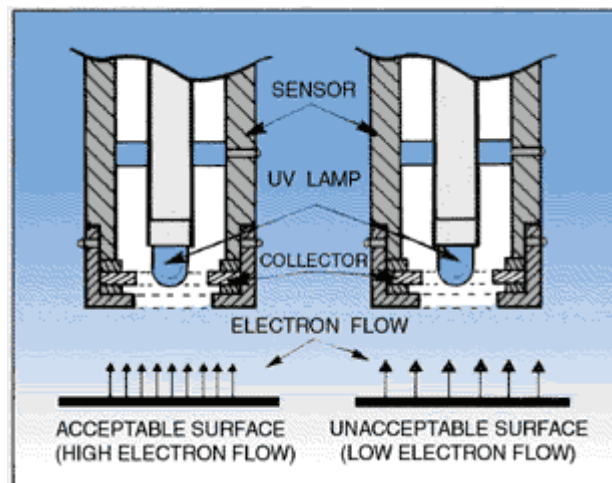


Figure 3 Cross-sectional diagram indicating the principle of operation for the SQM-200 (<http://www.photoemission.com/>)

2.2.1 Reference plate measurements

Surface quality measurements were taken on 6" by 6" clad Al2024-T3 aluminium alloy plates at various stages of the surface abrasion and grit-blasting steps using a specially manufactured X and Y scanning stage and associated control software (Figure 4). Maps of 160 by 160 pixel resolution were acquired on the aluminium plates in 2 to 3 minutes using a step size of 10 mm. As previous work had indicated that the SQM signal dropped rapidly with time, the two dimensional map was the average of 2 scans, with the second scan beginning at the final location of the first scan. The averaging enabled the signal at the same point over a period of around 2 minutes to be recorded. The gain setting of the

detector was 2 and the distance between the head and surface was 2 mm. An aperture of 3.15mm diameter was used.

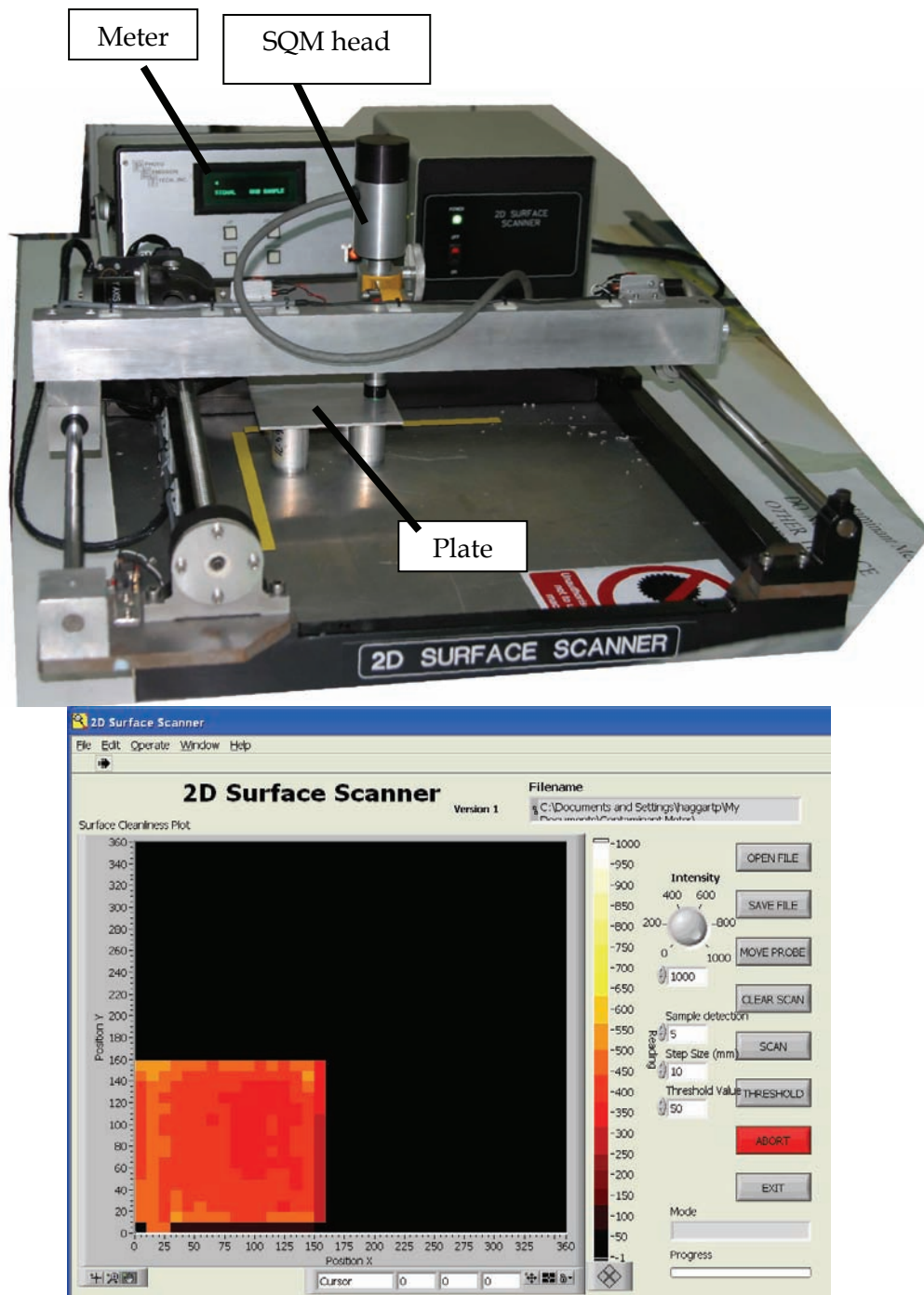


Figure 4 The two dimensional surface scanning unit and software acquisition screen used to map the SQM-200 output from 6" by 6" aluminium plates surface treated for bonding.

2.2.2 Measurement of plates before bonding

As indicated in section 2.1.3, previous work produced a series of wedge tests in which several important parameters were adjusted. During the fabrication of the wedge test plates, a series of Surface Quality Monitor measurements were taken during the grit-blasting and silane application stage of the surface treatment procedure. This provided a direct correlation between surface quality and bond quality, as measured by the wedge test. Unlike the two dimensional maps acquired in section 2.2.1, measurements of the 6" by 6" wedge test plates were acquired at 3 locations in the central section of each plate (Figure 5), using a gain setting of 2, a 2 mm measurement height and a 3.15 mm diameter circular aperture.

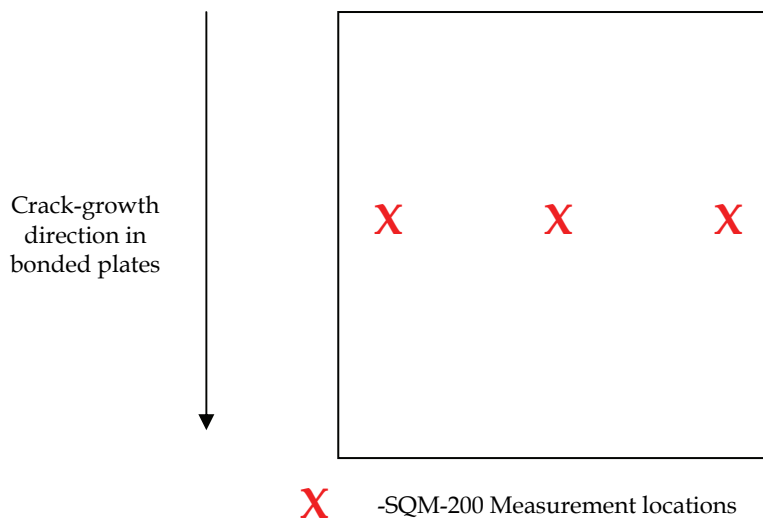


Figure 5 Measurement locations on the clad Al2024-T3 aluminium plates for the SQM-200 after grit-blasting and silane treatment during wedge test manufacture.

3. Results

3.1 Gloss-meter measurements

3.1.1 Reference plate measurements

Figure 6 shows the gloss units measured for clad Al2024-T3 aluminium plate after solvent cleaning and abrasion. The results are presented for measurements parallel and perpendicular to the grain direction and were the average of the 60 degrees measuring direction. Measurements for the three individual angles are provided in Appendix B: . The large error for the MEK cleaned surface was associated with changes for reflectivity as the measurement height increased (Figure 23). After abrading the plate, the gloss units

dropped from around 550 to less than 150. The decrease in gloss units after abrasion was also dependent on the orientation of the abrasion direction with respect to the measurement direction of the gloss-meter. When measurements were conducted with the gloss-meter in the same direction as the abrasion, values between 110 and 150 gloss units were recorded. However, for measurements made normal to the abrasion direction the gloss units dropped below 50. Whilst these changes were present for all the measuring angles, Figure 24 to Figure 28 in Appendix B: highlight that at 60 degrees these differences were substantially greater. Clearly, the abrasion in the measurement direction assisted in guiding the reflected light to the detector and this was most effective for the 60° reflection angle. Presumably, when measurements were made normal to the abrasion direction the reflected light was scattered away from the detector, reducing the amount of detectable light. Interestingly, differences in gloss units for the different levels of abrasion; light, medium and heavy, did not appear to be a major factor that influenced the gloss unit levels, suggesting that even light abrasion significantly alters the surface reflectivity.

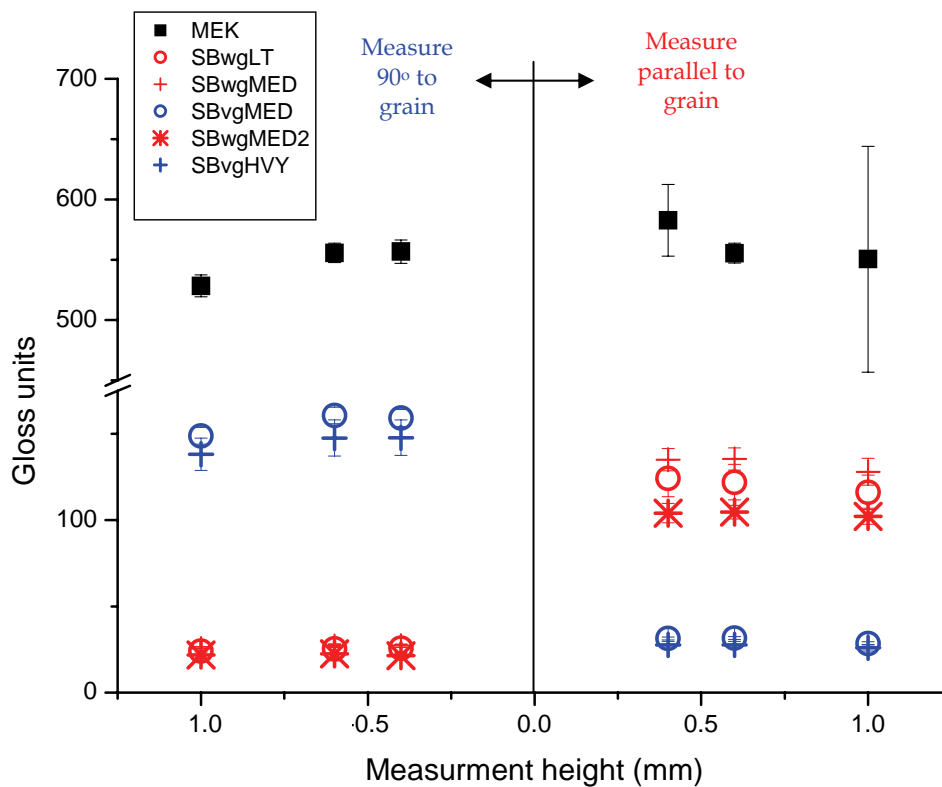


Figure 6 Gloss units as a function of measurement height for MEK solvent cleaned and Scotchbrite abraded Al2024-T3 clad aluminium alloy plates: ■ MEK cleaned, ○ light abrade with grain, +, * medium abrade with grain, ○ medium abrade against the grain, + heavy abrade against the grain.

Figure 7 shows the gloss unit values for clad Al2024-T3 aluminium plate after solvent cleaning or abrasion and followed by grit-blasting. Compared with Figure 6, the grit-blasting has substantially reduced the gloss of the solvent cleaned or abraded metal surface. The results suggest that the grit-blasting was not obviously affected by the condition of the metal prior to the grit-blasting step, at least within the variation observed for 30 individual measurements at each angle. Figure 7 shows that the 60 degree angle was relatively insensitive to height with a small increase in gloss unit values occurring at 0.55 mm and 0.35 mm heights. However, results for individual measurements made at 20, 60 and 85 degrees shown in Appendix B: shows that at 85 degrees the gloss unit reading increased substantially when the measurement was made closer to the surface.

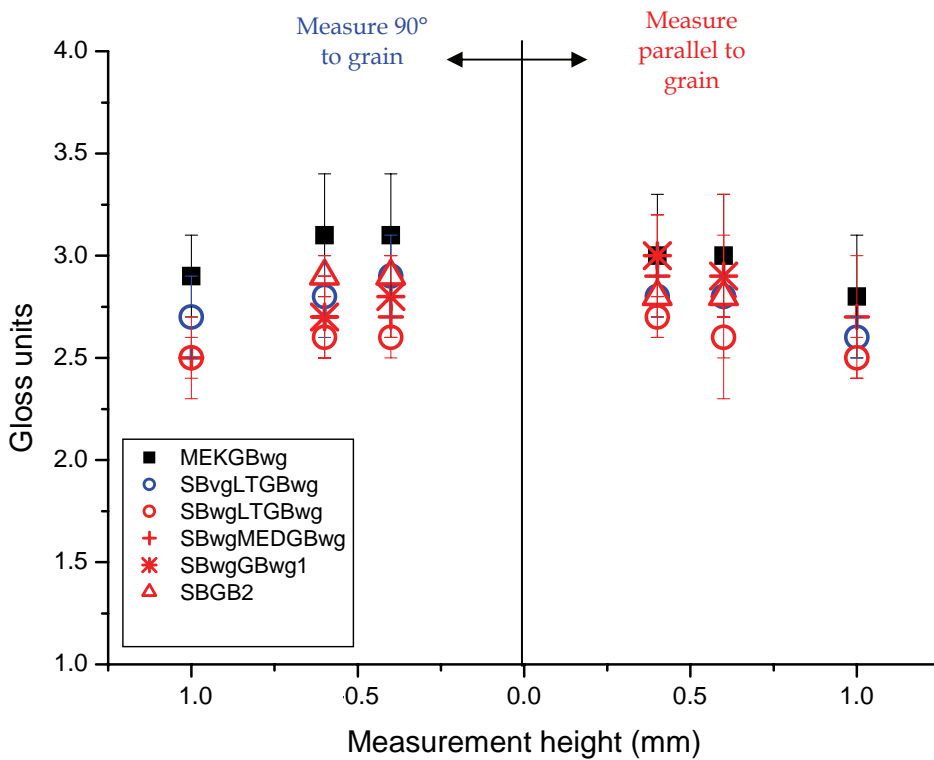


Figure 7 Gloss units as a function of measurement height for Al2024-T3 clad aluminium alloy plates after MEK solvent cleaning or Scotchbrite abraded followed by grit-blasting: ■ MEK cleaned and grit-blasted with grain, ○ light abrade against grain and grit-blasted with grain, ○ light abrade with grain and grit-blasted with grain, + medium abrade with grain and grit-blasted with grain, * abrade with grain and grit-blasted with grain, △ abrade and grit-blast.

Whilst the values plotted in Figure 6 and Figure 7 are the average of 30 measurements for each data point, the standard deviations, shown as error bars, are relatively small, at least for the abraded and grit-blasted surfaces. A typical plot of the gloss unit readings across a

6" by 6" grit-blasted plate in Figure 8 shows that values are relatively constant over the plate for the three heights using the 60° measurement angle.

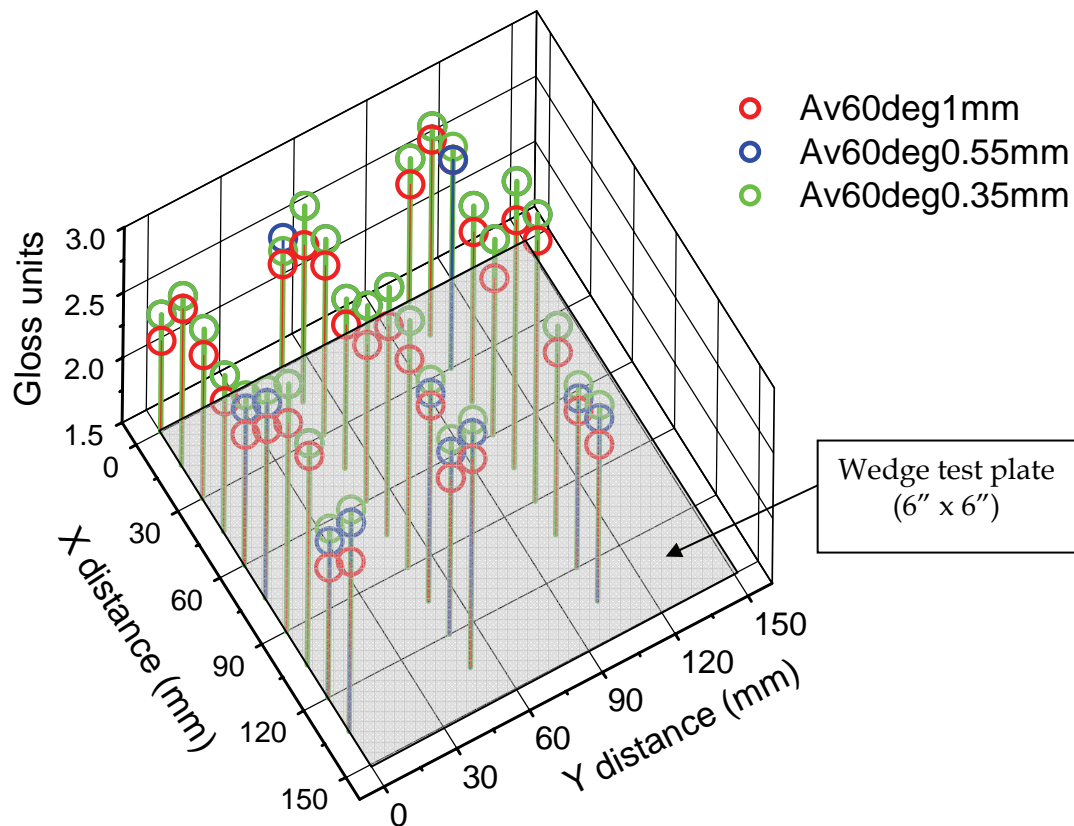


Figure 8 Individual measurements taken at 60 degrees and parallel to the grain direction for Al2024-T3 clad aluminium plate after MEK wipe and grit-blasting at three measurement heights, 1mm (red), 0.55mm (blue) and 0.35mm (green).

A summary of the spread in gloss unit readings for solvent cleaned, abraded and grit-blasted clad Al2024-T3 aluminium plates is shown in Figure 9. This provides an indication of the range of gloss units that would be expected for an aluminium plate that was solvent cleaned, abraded and grit-blasted by a trained technician using appropriate equipment and using a measurement height between 0.3 mm and 0.8 mm. Whilst orientation of the abrasion direction for the surfaces that are only abraded had a significant influence on the gloss readings, once grit-blasting was completed, gloss unit values converged to a value around 2.8. Previous work that looked at optimising the wedge test results for a grit-blast and silane surface treatment [8], indicated that a reading around 2.8 gloss units provided an optimum grit-blast. These results indicate that with the use of the gloss-meter unit, a trained technician should be able to reproducibly provide an optimum grit-blast for bonding.

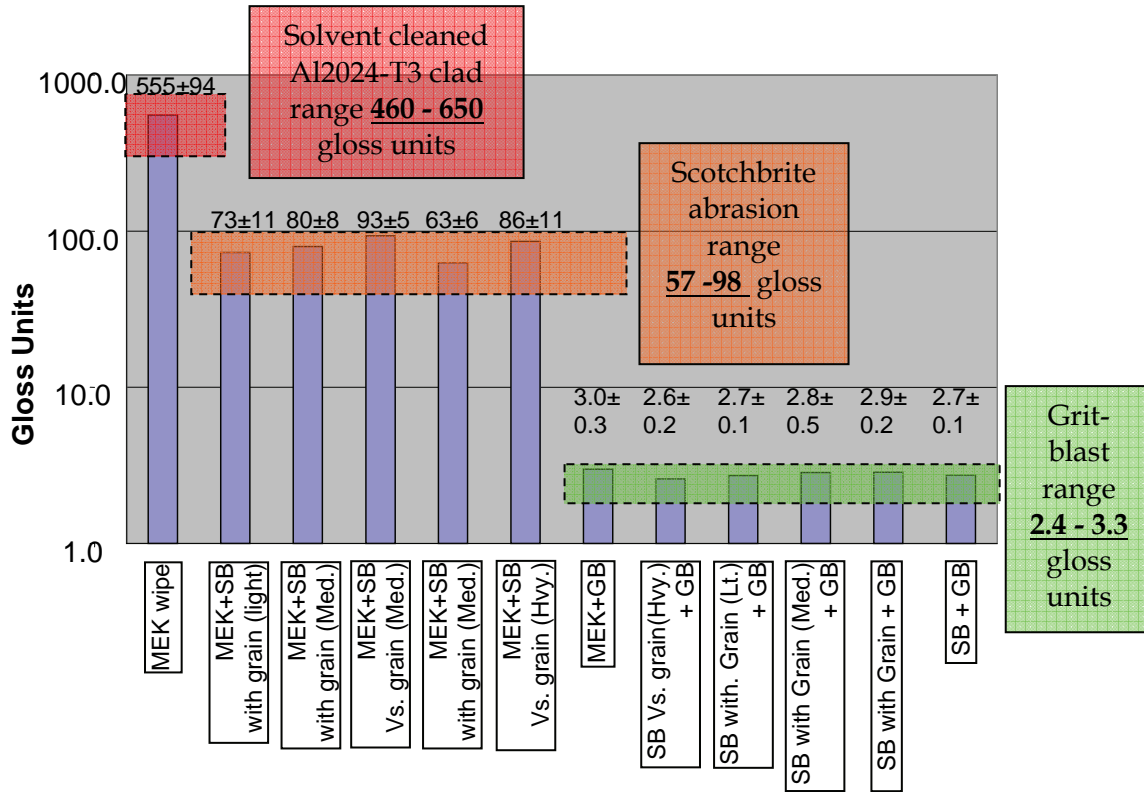


Figure 9 The range of gloss units measured at a 60 degree reflection angle and three stand-off heights of 0.7, 0.55 and 0.35mm for Al2024-T3 clad aluminium surfaces. The various stages of the aluminium surface treatment from solvent cleaning to abrasion and finally grit-blasting show variations due to the abrasion direction with respect to the measurement orientation.

3.1.2 Measurement of BSTT wedge test plates

Figure 10 shows the range of gloss-meter readings from grit-blasted wedge test plates produced during testing and requalification of bonding technicians by the Bonded Structures and Testing Team (BSTT). Statistical analysis of the plot reveals that the average gloss-meter reading corresponded to 2.8 ± 1.1 gloss units for a corresponding crack-growth of 41.7 ± 3.8 mm after 48 hours humid exposure at 50°C for the wedge test specimens. The gloss unit average value was very similar to that achieved by the trained technician in section 3.1.1, although the standard deviation was somewhat higher. Figure 10 shows that most data was clustered around 3 gloss units, however, a few outliers existed, which would contribute to the higher standard deviation. The reason for the outliers is unclear, but may be due to faulty equipment, which has been reported by BSTT previously in some instances where grit-blasting units had caused problems during the testing.

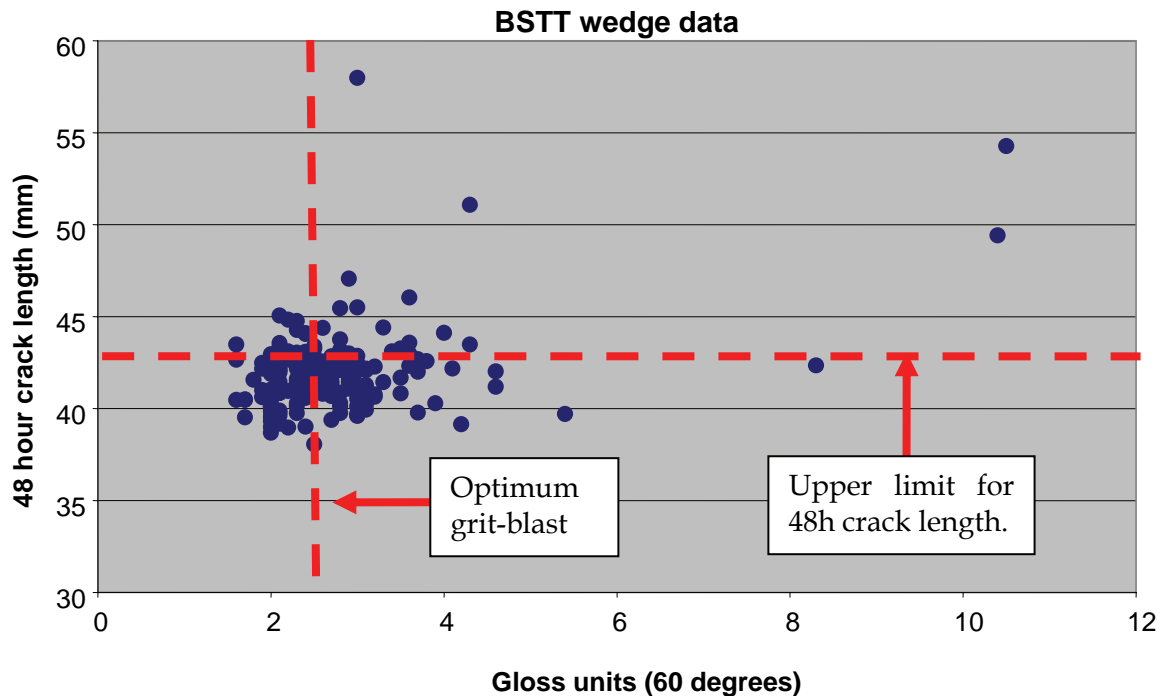


Figure 10 Gloss-meter readings for clad Al2024-T3 aluminium alloy plates that have been grit-blasted during training and requalification testing of ADF bonding technicians by BSTT.

3.1.3 Measurement of plates before bonding

A series of wedge test plates were manufactured as a part of the development of a regression model to optimise the current RAAF grit-blast and silane surface treatment for bonding thermoset epoxy adhesives to aluminium [8]. During the optimisation studies 50 wedge test panels were produced and as a part of the manufacturing process the panels had the grit-blast quality measured using the gloss-meter (Figure 11). Batch number 1 represented typical data when the technician was attempting to provide a standard grit-blast treatment, whereas, batches 2 and 3 show the spread when the technician was attempting to produce “lighter” (>3.5 gloss units) or “heavier” (<2.0 gloss units) grit-blasting. Even in the case where other variables were being adjusted during the wedge test manufacturing process, it can be clearly seen that in the range of 2 to 3 gloss units a small spread in crack lengths after 48 hours exposure to a humid environment was produced. For batch one measurements, which represent typical variation for an experienced and inexperienced operator, grit-blast surfaces could be produced with a reading of 2.6 ± 0.2 gloss units. This value was similar to those reported for reference plates measured from different heights in section 3.1.1 and 3.1.2 for the BSTT testing.

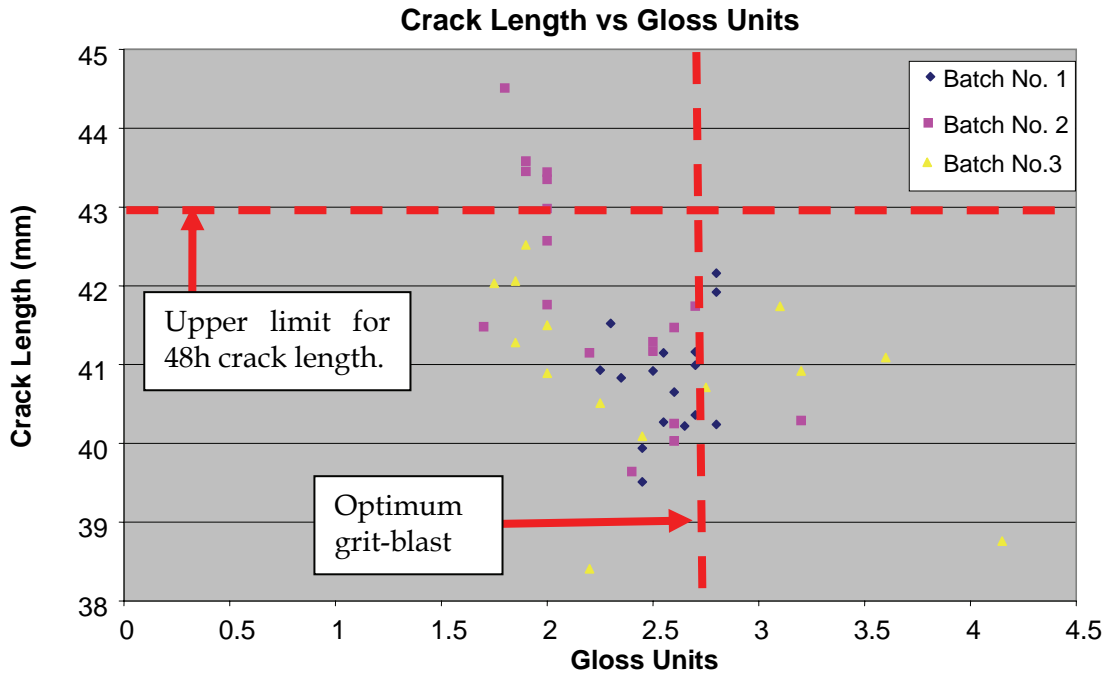
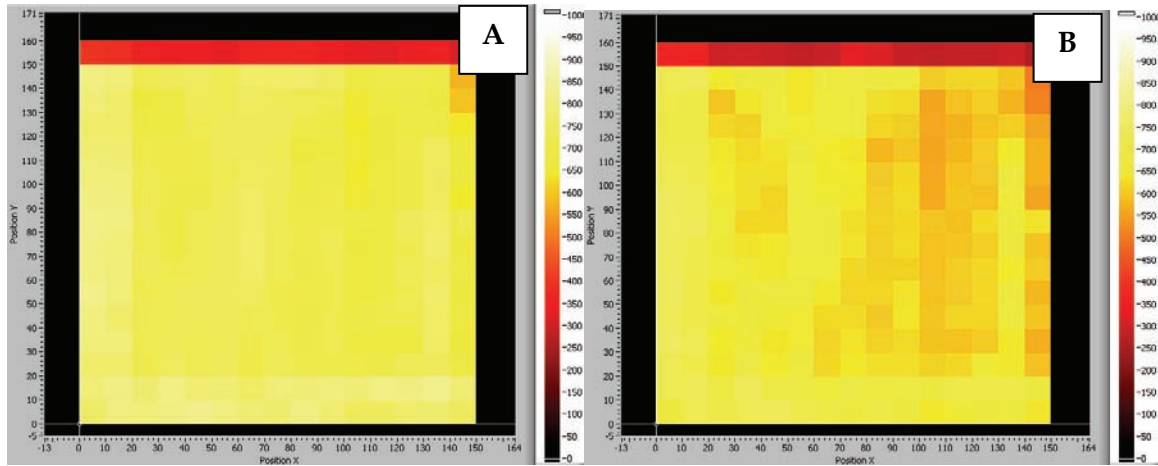


Figure 11 Average gloss units measured at a 60 degree angle for clad Al2024-T3 aluminium alloy plates directly after grit-blasting and prior to bonding with FM300 adhesive. Crack length is measured for the wedge test after 48 hours in a 50°C/95% RH environment.

3.2 Surface quality measurements

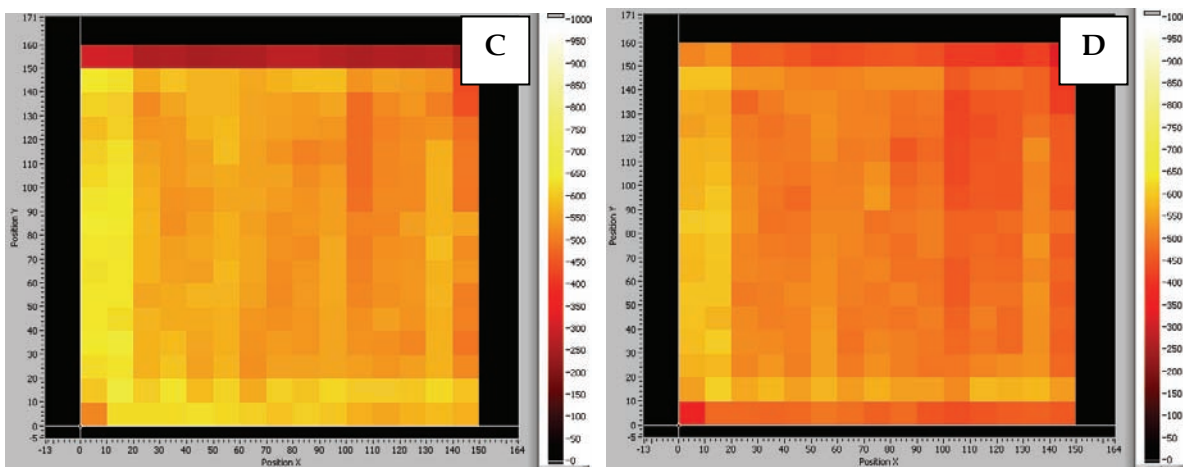
3.2.1 Grit-blasted reference plate measurements

Figure 12 shows the maps acquired for 6" by 6" clad aluminium plates after grit-blasting. Generally, the signal was quite uniform and decreased in intensity with exposure time to the laboratory environment, which was typically 21°C and 55 to 65% relative humidity (R.H.). The decline in signal recorded by the SQM-200 for 5 individual plates that were grit-blasted is shown in Figure 13. The results are plotted as the average signal from the maps of the 6" by 6" plates with the error bars representing the standard deviation in the signal across the mapped region.

Gloss units: 2.4

Mean=732±43

Mean=643±47



Mean=558±42

Mean=500±45

Figure 12 SQM-200 scan using 10mm steps over an area of 160mm by 160mm for a grit-blasted plate. Images were acquired at (A) 15 min. (B) 45 min. (C) 100 min. (D) 150 min. after the initial grit-blasting. The reading scale is from 0 (black) to 1000 (white). Mean readings and standard deviations are also reported.

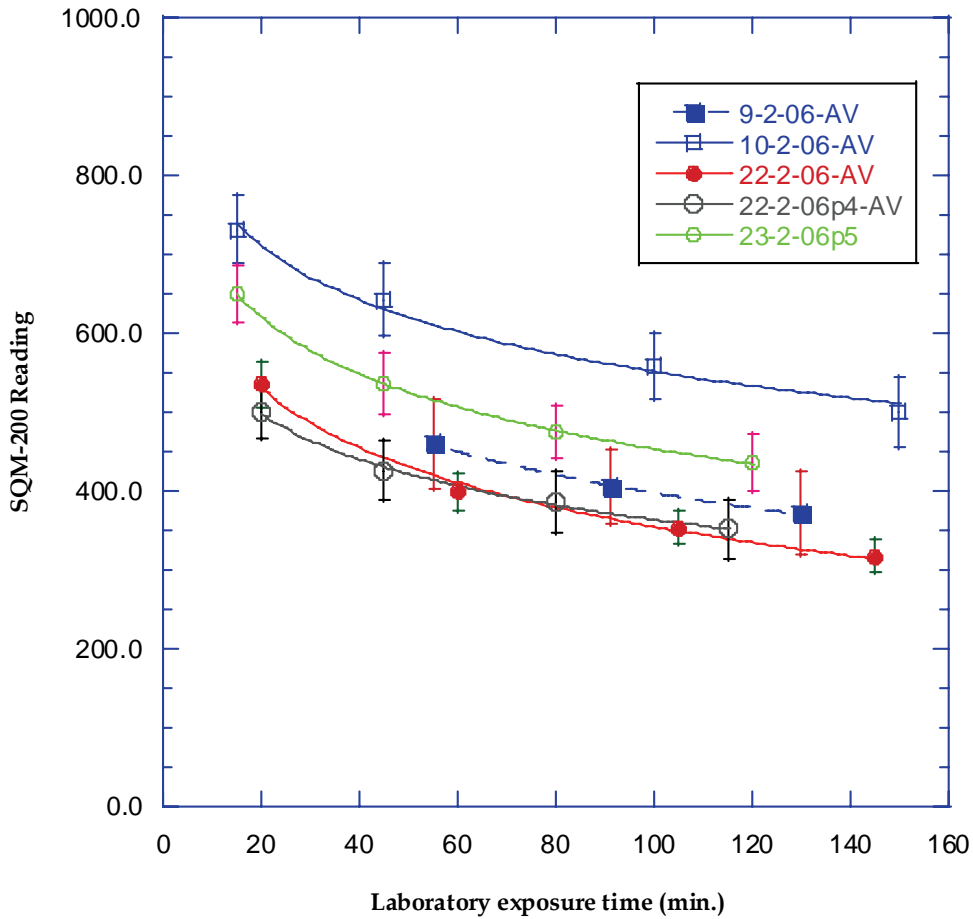


Figure 13 SQM-200 average reading with time over a grit-blasted 6" by 6" clad Al2024-T3 aluminium alloy plate after a 3 minute scan using a 10 mm step size.

Figure 13 shows the average signal over a 6" by 6" clad Al2024-T3 aluminium alloy plate after grit-blasting. Slopes and intercepts for logarithmic fits to the data are provided in Table 2. The logarithmic fits suggest that at zero exposure time, directly after grit-blasting was completed, the SQM-200 signal could be around 900 and this signal could drop as low as 300 over a 2 hour period (Table 3).

Table 2 Logarithmic fit to the data plotted in Figure 13, indicating the SQM-200 reading at time zero (Y intercept) and the signal reduction rate (log(X)).

Surface Scan	Y intercept	Gradient log (X)	Fit Coefficient
9-2-06	870	-237	0.999
10-2-06	1008	-228	0.993
22-2-06	858	-251	0.997
22-2-06p4	747	-192	0.999
23-2-06p5	929	-238	0.999
Average	882	-229	---
Std Dev.	96	22	---

Table 3 Average SQM-200 reading with time in a lab environment for grit-blasted Al2024-T3 clad aluminium alloy.

Exposure time after grit-blasting(min.) 21°C/55-65% R.H.	SQM reading on grit-blasted aluminium
0	882
20	653
30	543
60	475
120	405

3.2.2 Measurement of grit-blasted plates before bonding

Figure 14 shows the trend in SQM-200 readings as a function of gloss units for aluminium plates that had been grit-blasted during surface treatment of wedge test samples for the development of a regression model. Most of the results for the three batches of testing showed SQM values around 500-600 for gloss units around 2.5. There appeared to be a weak trend for lighter grit-blast surfaces (gloss units above 3) to have the SQM readings drop below 500, although one reading below 400 was also observed at 2.0 gloss units.

Figure 15 shows the 48 hour crack length for wedge test samples manufactured for the development of the regression model and the corresponding SQM-200 reading measured after the grit-blast step. Error bars in Figure 15 are the standard deviation in the SQM-200 readings for the two plates measured at 3 locations for each plate. The SQM-200 readings were clustered around 500-600 and the corresponding crack lengths at 48 hours were between 41 and 43 mm.

Gloss units vs SQM-200 Reading

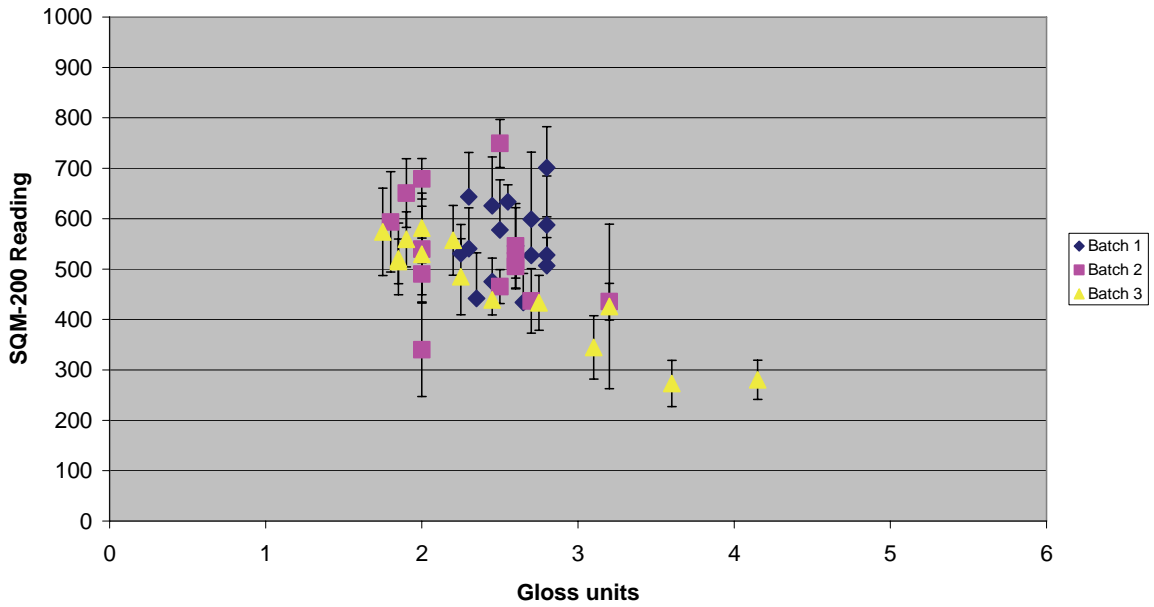


Figure 14 SQM-200 measurements taken on grit-blasted clad Al2024-T3 aluminium plates used for wedge test manufacture plotted as a function of gloss units.

SQM-200 Readings vs Crack Length

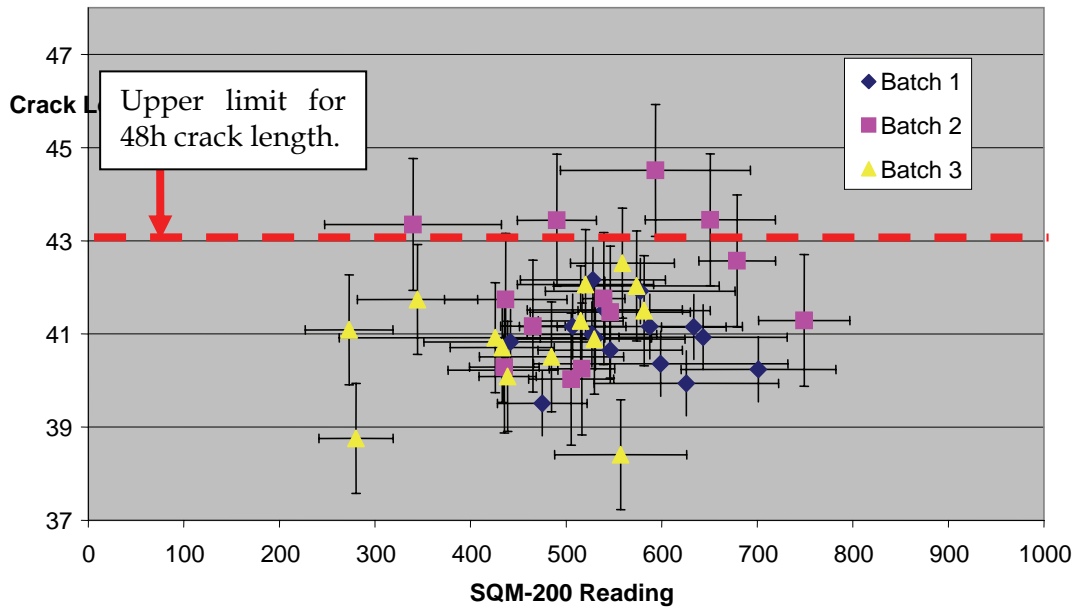


Figure 15 Crack length measured after 48 hours humid exposure for wedge test samples where the grit-blast surface was measured with the SQM-200 prior to bonding.

Figure 16 shows the average SQM-200 readings for the grit-blasted surfaces for the wedge test plates produced during the development of the regression model. Within the error bars, the average value appeared to be around 500 for the three batches. The measurement represented an average value for the two plates taken around 20 to 30 minutes after the grit-blasting was completed. This value was within the range estimated in Table 3 for the 6" by 6" plates that were scanned. This result suggests that a lower limit could be set as a minimum requirement for an acceptable surface cleanliness state after grit-blasting.

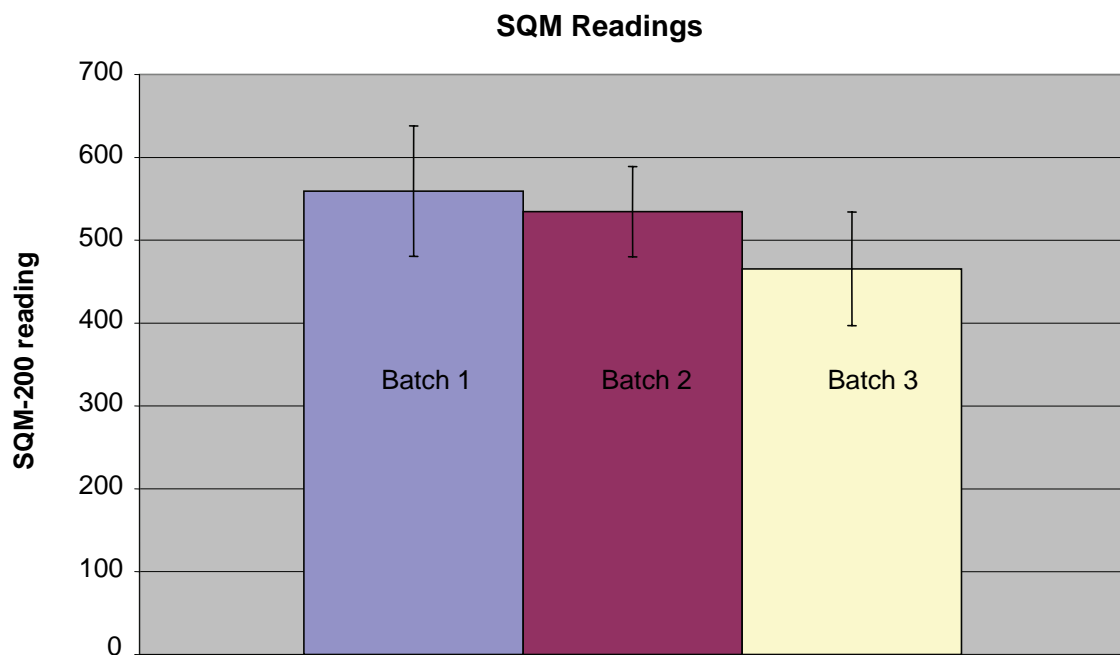


Figure 16 Average SQM-200 readings for the 3 batches of wedge test specimens manufactured during the development of the regression model.

3.2.3 Grit-blasted and Epoxy Silane treated plates

Figure 17 shows the average SQM-200 values after epoxy silane was applied to grit-blasted aluminium plates during the manufacture of wedge test samples for the development of the regression equation. A substantial variation in the results appeared due to a number of variables that were examined during the wedge test manufacture. The variables included changing the silane concentration from 0.5 to 2% v/v, altering the grit-blast level from 1.7 to 3.6 gloss units and changing the method of drying from nitrogen removal to heat gun drying.

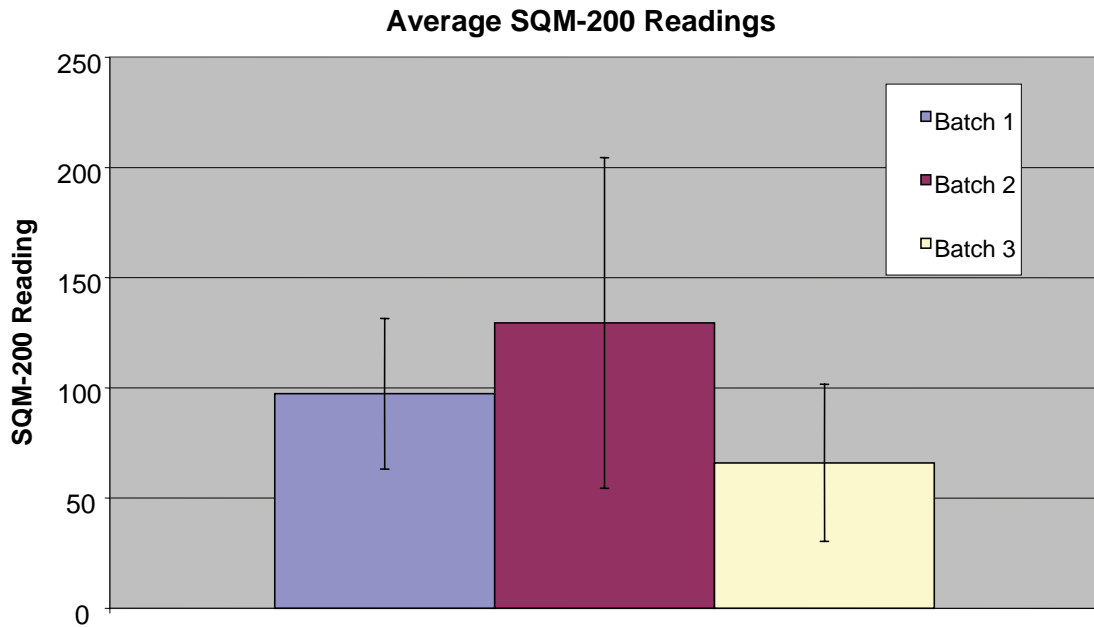


Figure 17 *Average SQM-200 readings for the three batches of wedge tests manufactured for the development of the regression model after application of epoxy silane between 0.5 and 2% v/v solution.*

The trends of each of these variables with the SQM-200 readings are shown in Figure 18 (% silane), Figure 19 (grit-blast level) and Figure 21 (drying method). The general trend observed in Figure 18 was the decrease in SQM-200 reading with increasing silane concentration. This may be expected as a thicker silane layer would attenuate the photoelectron signal. Figure 19 shows a weak trend between gloss units and SQM-200 readings. Generally, as the gloss units increased (or the level of grit-blasting became lighter) the SQM-200 reading decreased. This trend was consistent with the pre-existing grit-blasted surfaces shown in Figure 14. Figure 20 shows the correlation in SQM-200 readings for the grit-blasted surface before and after silane treatment. There was a weak trend that showed that the initial grit-blast reading influenced the reading after silane treatment, with higher values being recorded on silane treated surfaces where the original grit-blast reading was high.

The final trend in Figure 21 suggests that the SQM-200 reading generally decreased for the heat gun drying method. This trend may be explained in terms of the heat gun either leaving a thicker layer of silane on the surface or localised heating of the aluminium increasing the oxide layer thickness. In either case, the photoelectron signal would be attenuated.

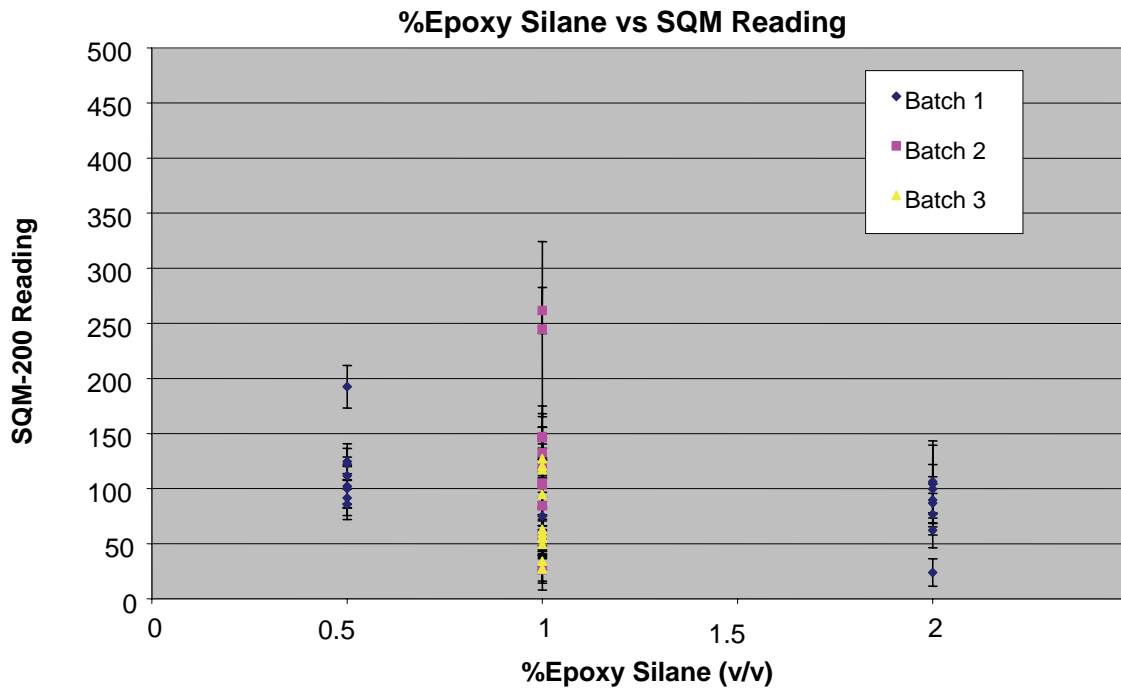


Figure 18 SQM-200 readings as a function of the epoxy silane solution concentration applied to the grit-blasted aluminium wedge test plates

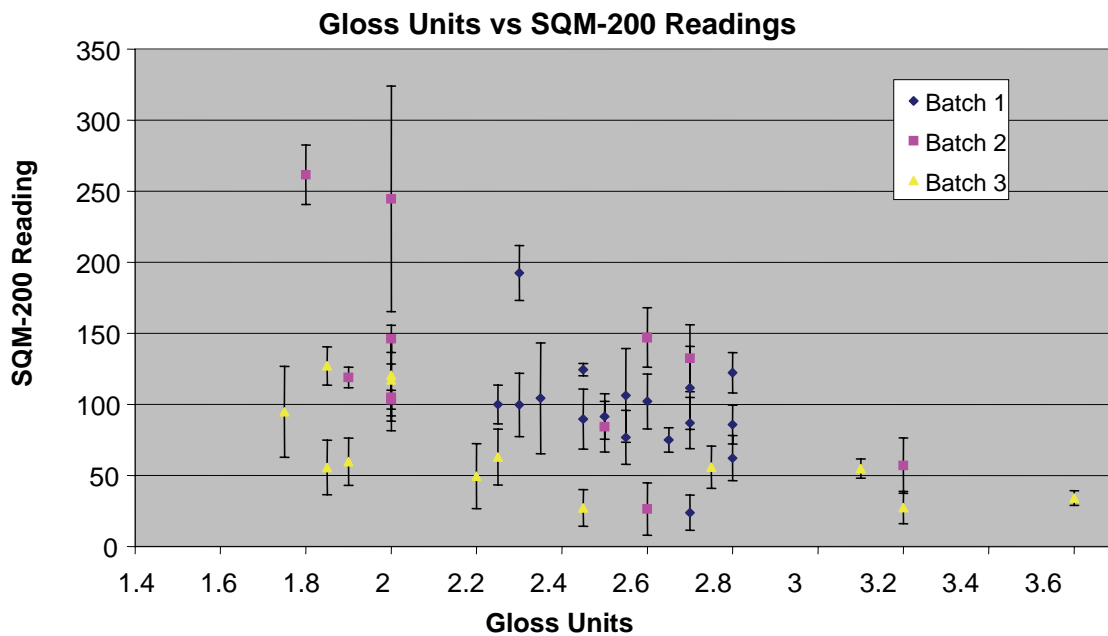


Figure 19 SQM-200 readings for the different grit-blasted and silane treated surfaces that were characterised by gloss units measured with the gloss-meter prior to silane treatment.

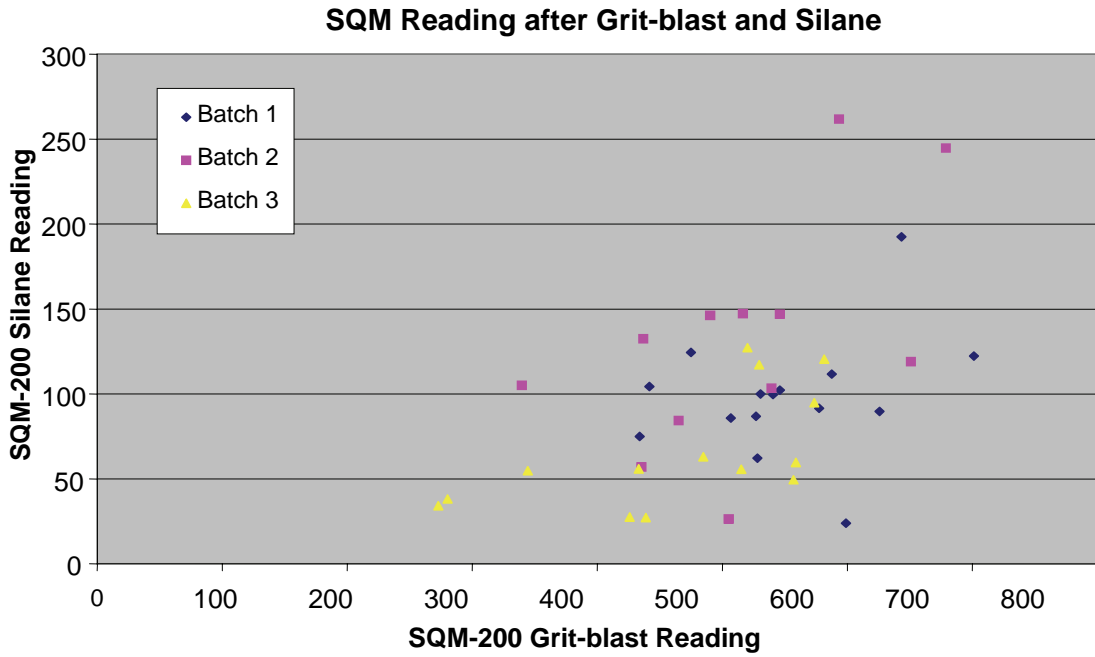


Figure 20 The correlation in SQM-200 readings for grit-blasted aluminium plates before and after treatment with a 1% epoxy silane solution.

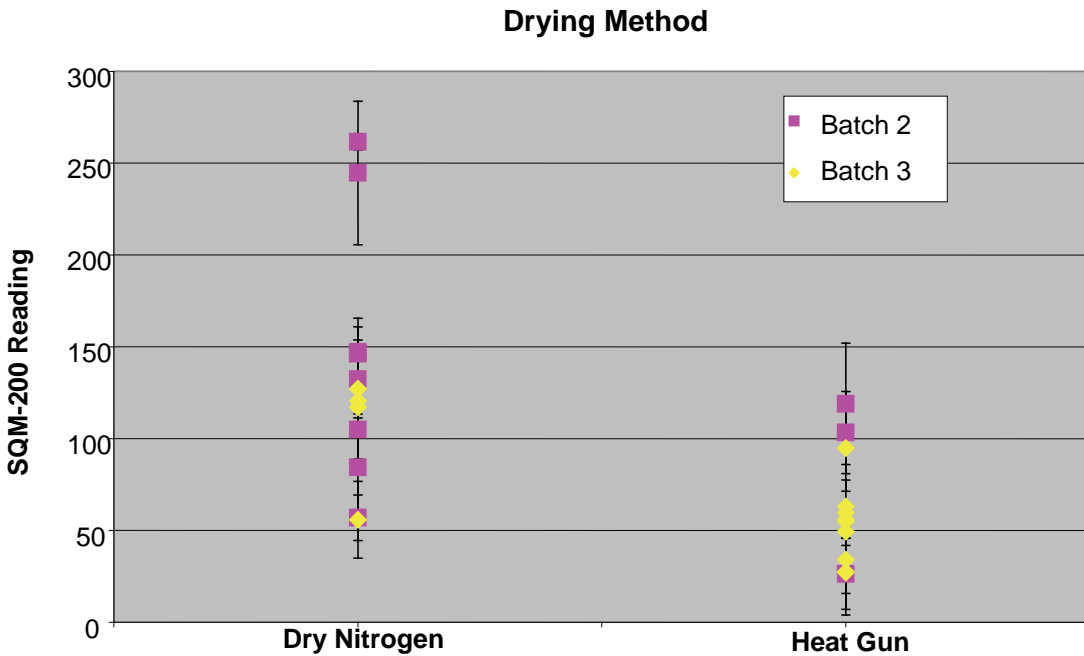


Figure 21 SQM-200 readings for grit-blasted and silane treated aluminium plates dried using either a nitrogen stream or a hot air gun

The crack length at 48 hours exposure as a function of SQM-200 readings for the wedge tests manufactured for the development of the regression model are shown in Figure 22. Due to the level of variation in the results it is difficult to establish any trend in the data, with most values falling near 41 to 42 mm and 90 to 120 for the SQM-200 reading. Clearly, further experimental work would be required to isolate the various sources contributing to the change in SQM-200 signal on the silane treated surfaces.

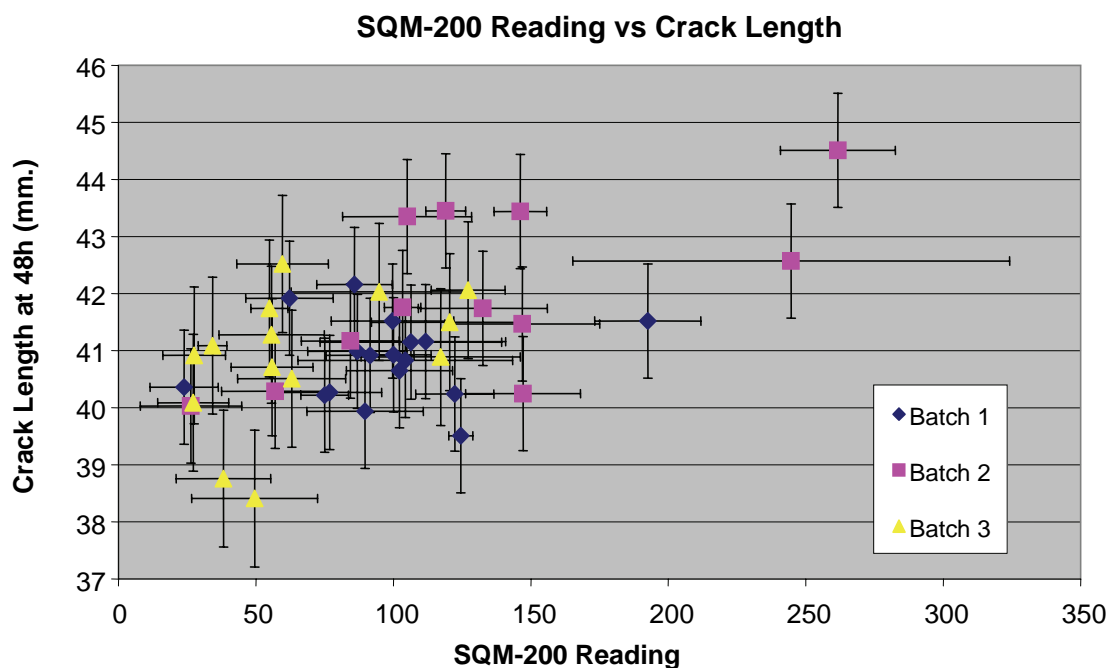


Figure 22 Crack length at 48 hours exposure as a function of SQM-200 readings for grit-blasted and epoxy silane treated aluminium wedge test plates.

4. Discussion

4.1 Gloss-meter measurements

Figure 6 shows that the gloss-meter can clearly discriminate between different levels of surface roughness produced on aluminium plates as a result of abrasion or grit-blasting. The reduction in gloss units from the as received aluminium surface to the abraded and grit-blasted surfaces revealed that the surface became progressively less reflective as the aluminium plate was processed with a typical grit-blast and silane surface treatment. Interestingly, the reflection of light was sensitive to the direction of abrasion, with

scratches from abrasion made normal to the measurement direction substantially reducing the amount of reflected light that could be detected. Despite the sensitivity of the reflected signal to abrasion direction, the subsequent grit-blasting process appeared to not be sensitive to the direction of measurement, suggesting that, if the grit-blasting was performed correctly, then a homogenous surface roughness was generated. The sensitivity of the gloss-meter reading to abrasion direction would, however, become an important parameter if grit-blasting was too light and the abrasion step was still contributing to the level of reflected light.

Figure 7 shows that the gloss-meter could be successfully applied in non-contact mode without any substantial changes to the measured signal resulting, at least for the signal measured at 60 degrees. Variation in the measured grit-blast signal appeared to be more sensitive to the ability of the technician to undertake the operation in a repeatable manner than either the measurement height, direction or level of abrasion performed before the grit-blasting. Nevertheless, variation in the gloss units measured for seven separate plates appeared to be relatively small when the grit-blasting was undertaken by a trained technician (Figure 9). Additionally, a range of measurements taken over the whole of a 6" by 6" plate suggested that the grit-blasting could also be performed uniformly (Figure 8).

Confirmation of the ability of trained technicians to undertake the grit-blasting step satisfactorily were provided by assessment of the gloss-meter readings measured for a large population of technicians who produced wedge test plates during training and requalification by BSTT. Most technicians achieved a gloss-meter reading around 2.8, with the standard deviation being skewed by some particularly high readings, where grit-blasting had not been performed properly (Figure 10). Around 2.8 gloss units also provided a small distribution in crack length values measured after 48 hours for wedge tests, confirming the regression model value, which indicated an optimal grit-blast was around 2.8 gloss units. Wedge test panels produced during the development of the regression model also confirmed that when an experienced and inexperienced technician were attempting to produce a uniformly grit-blasted surface that a value around 2.6 gloss units was achieved with very small deviation (Figure 11).

Studies with the gloss-meter unit confirm that using a measurement angle of 60 degrees and a measurement height around 0.5 mm that reliable measurements of the grit-blasting process could be made. Correlation in the gloss units measured for the grit-blasted surface and the 48 hour crack length for the subsequently produced wedge test samples confirmed that adhesive bonding could be performed reliably and reproducibly.

4.2 Surface Quality measurements

The SQM-200 indicated that it was very sensitive to the surface condition of grit-blasted aluminium wedge test plates. After grit-blasting the plates, signal decreased in a logarithmic manner with time of exposure to the laboratory environment. The decrease in measured signal was a combination of oxide layer thickening and surface contamination, both processes increased the overlayer thickness of the metal surface and consequently attenuated the photoelectron signal. The use of a scanning frame and a signal processing algorithm, however, was very successful at providing a reliable image of the surface

cleanliness. The results indicated in Figure 12 clearly showed that the grit-blasting could provide a uniform surface condition.

Despite the reduction in signal, measurements conducted on 5 individual wedge test plates revealed that the signal reduction was predictable and an upper signal limit could be set that would establish a minimum surface cleanliness condition that was acceptable for proceeding with the next step in the bonding operation. Based on the averages shown in Table 3, it would be possible to set a time limit between the grit-blasting step and the application of the epoxy silane solution, or a minimum signal requirement. Based on the current standard [4], the recommended exposure time for a laboratory, is around 30 minutes, which would correspond to a signal around 500 from the SQM-200 (Table 3).

A minimum SQM-200 reading around 500 after grit-blasting appeared to be a reasonable value as a cut-off point. The average readings measured around 30 minutes after treatment for wedge test panels manufactured for the development of the regression equation (Figure 14) showed values clustered around 500. The SQM-200 reading was also sensitive to the level of grit-blasting and when gloss units around the 2.6 value were achieved the SQM-200 readings also averaged around 500 (Figure 14). As the level of grit-blasting decreased and gloss units increased, SQM-200 readings also decreased. This showed the importance of combining the gloss-meter readings with the SQM-200 readings to provide reliable levels of surface cleanliness on grit-blasted aluminium plates. Further support for using an SQM-200 value around 500 as a minimum surface condition for a grit-blasted surface was provided by a correlation between wedge test 48 hour crack lengths and SQM-200 readings after the grit-blast step. Figure 15 shows SQM-200 readings clustered around 450-550 provided a narrow distribution of crack lengths.

The use of the SQM-200 was also applied to characterising the surface condition of the grit-blasted aluminium plate after treatment with epoxy silane. Deconvolution of the wedge test panel measurements made on epoxy silane treated surfaces prepared for the regression model revealed that the SQM-200 readings were sensitive to a number of parameters. The parameters included epoxy silane solution concentration (Figure 18), grit-blasting level (Figure 19) and drying method (Figure 21). Whilst the average readings were quite variable (Figure 17), weak trends in SQM-200 readings with the mentioned parameters could be established. SQM-200 signal decreased with increasing silane concentration and when the silane was dried using a heat gun, compared to nitrogen gas. Both these changes may have been related to increased overlayer thicknesses attenuating the photoelectron signal. Higher silane concentrations may be expected to produce thicker films and heat gun drying of the films would also accelerate oxide growth. A weak trend with gloss units and SQM-200 readings was also observed after silane treatment, with the SQM reading decreasing as the gloss units increased. Increased gloss units represent a surface with a lighter grit-blast existed prior to the application of the silane solution. This trend was similar to that observed for the pre-existing grit-blasted surface observed in Figure 14 and showed that the SQM-200 value measured after silane treatment was, to some extent, dependent on the cleanliness of the grit-blasted surface.

Generally, it would appear that the SQM-200 could be applied to characterise surface cleanliness of grit-blasted aluminium plates and provide a minimum value that could be

used as an indicator of whether the next step in the surface treatment process could proceed. Application of the SQM-200 to measure the epoxy silane treated surface, however, had a number of contributing factors and would require more investigation to provide a reliable measure of the effectiveness of the silane treatment prior to bonding.

5. Conclusions

- 1) The Micro TRI Gloss gloss-meter unit can be used to accurately measure the condition of abraded and grit-blasted aluminium plates using the 60 degrees measurement angle and from a measurement height of 0.5 to 0.7 mm.
- 2) Trained technicians can routinely produce a grit-blasted surface finish on clad Al2024-T3 aluminium plates with a reading of 2.8 gloss units.
- 3) Grit-blasting conducted by trained technicians can provide a homogeneous surface with minimal variation over a 6" by 6" square area.
- 4) Grit-blasting an aluminium plate to provide a finish with a reading of 2.8 gloss units will produce reliable adhesive bonded joints of good quality, as determined by the wedge test.
- 5) The SQM-200 is sensitive to the surface condition of grit-blasted clad Al2024-T3 aluminium plates and can be employed to establish surface contamination levels as a function of environmental exposure time.
- 6) If the SQM-200 reading is taken in conjunction with gloss meter measurements, then it is possible to provide a reliable assessment of the surface contaminant levels on grit-blasted clad Al2024-T3 aluminium plates.
- 7) SQM-200 readings around 500 measured 20-30 minutes after grit-blasting can provide a surface suitable for silane treatment and will produce reliable adhesive bonded joints of good quality, as determined by the wedge test.
- 8) SQM-200 measurements of epoxy silane treated grit-blasted aluminium surfaces are dependent on a number of factors and further work would be required to establish a reliable method of measuring the silane treated surface.

6. Recommendations

- 1) The Micro TRI Gloss gloss-meter unit should be used during training and requalification testing at BSTT to objectively measure the quality of grit-blasting performed by technicians and the measurements should be included in the QA Monitor database for each round of testing. A value around 2.8 gloss units using the 60 degree measurement angle and 0.5 to 0.7 mm measurement height should be the target value.
- 2) Methods to enable a 0.5 to 0.7 mm stand-off height for the gloss measurements should be investigated, including a jig for use in requalification testing and sapphire feet for field based repairs.

- 3) Bonded repairs conducted on primary aircraft structure should include a gloss-meter measurement of the grit-blasted surface during the repair and the value should be within the limits that guarantee a reliable adhesive bond of good quality, as measured by the wedge test.
- 4) The SQM-200 should be implemented in technician training and requalification testing conducted by BSTT to measure the surface condition after grit-blasting and used as a tool to determine whether the surface is adequate for continuing the surface treatment procedure.
- 5) Bonded repairs conducted on primary aircraft structure should include a SQM-200 measurement of the grit-blasted surface during the repair and the value should be within the limits that guarantee a reliable adhesive bond of good quality, as measured by the wedge test.

7. Acknowledgements

The author would like to thank Mr Peter Haggart (Fortburn Engineering) and Mr Peter Hales (Boeing Australia) for their significant technical contribution.

Appendix A: Grit-blast and silane surface treatment

The DSTO/RAAF surface preparation procedure included the following steps:

1. The plates were wiped with tissue using analytical grade methyl ethyl ketone (MEK) to remove gross contamination.
2. Scotch Brite 3M No. 447 pad was used to abrade the adherend surface with MEK. The surface was first abraded in one direction for 2 minutes then abraded in a direction 90° to the original direction until all original scratches were removed. The area was kept wet with MEK while abrading.
3. The adherend was wiped with MEK and tissues in the most recent direction (along the plate) to remove debris from the previous operation. This was continued until the tissues came up clean.
4. Distilled water and tissues were used to wipe the adherend surface again (along the plate).
5. Step 2 was repeated using distilled water in place of MEK.
6. Step 3 was repeated using distilled water in place of MEK.
7. The water break test was applied for 15 seconds to ensure a contaminant free surface. The adherend was held at 45° and a squeeze bottle was used to apply distilled water.
8. The adherend was dried in an air circulating oven for 20 minutes at 80°C.
9. The adherend was cooled to 35°C or less.
10. The bonding surface was grit blasted with 50µm aluminium oxide grit using dry nitrogen gas as a propellant, with a pressure of approximately 450 kPa.
11. The adherend was submerged in a one percent aqueous solution of γ -glycidoxypropyl trimethoxy silane (γ -GPS) for 10 minutes. This solution consisted of 1% γ -GPS + 99% deionised or distilled water. The solution had been stirred for at least 60 minutes prior to use.
12. The adherend was dried in an air circulating oven for one hour at 110°C.
13. The adherend was cooled to below 35°C.
14. The surfaces were bonded within 1 hour of being prepared to prevent contamination.

Appendix B: Gloss-meter readings for clad Al2024-T3

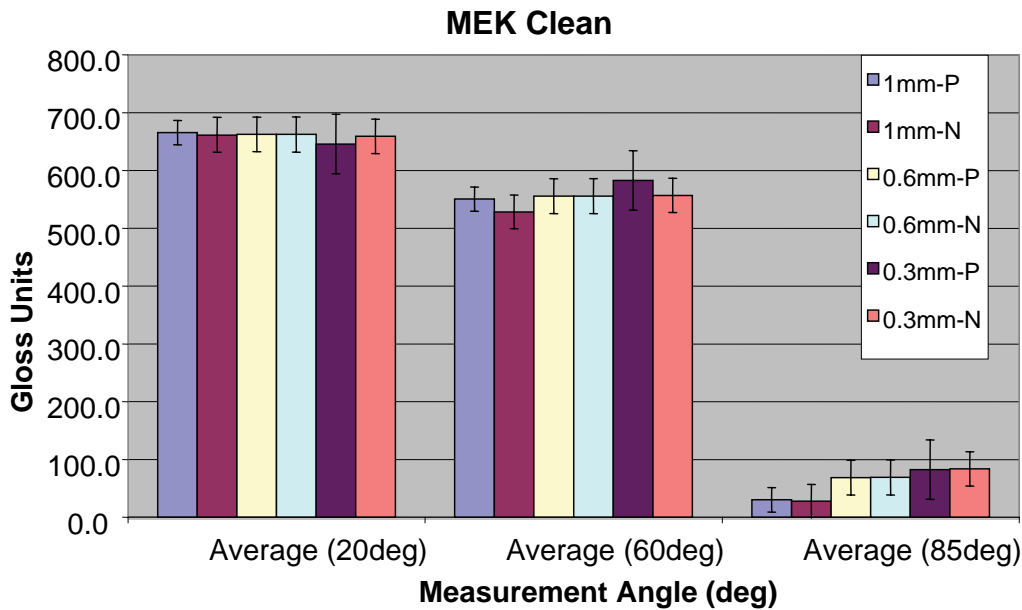


Figure 23 Gloss meter readings for MEK cleaned Al2024-T3 clad aluminium. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.

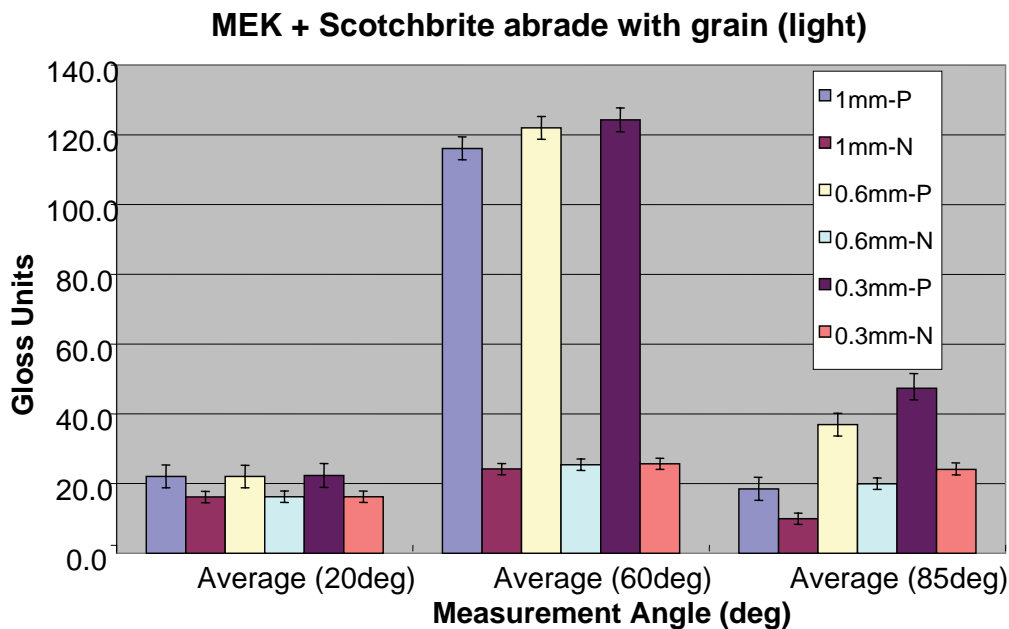


Figure 24 Gloss meter readings for Al2024-T3 clad aluminium that was MEK cleaned and lightly abraded in the grain direction. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.

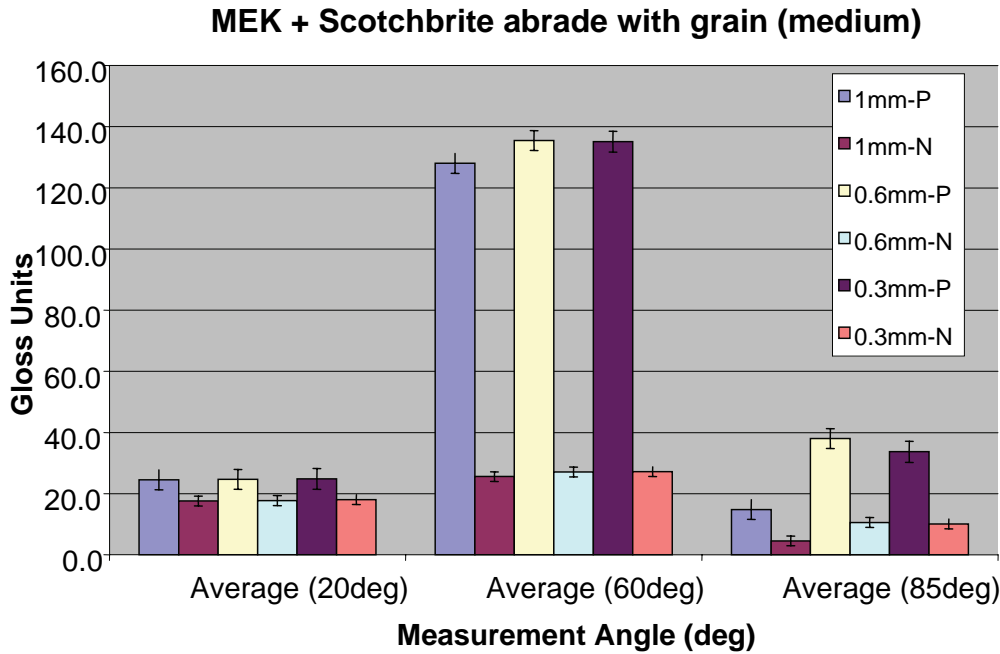


Figure 25 Gloss meter readings for Al2024-T3 clad aluminium that was MEK cleaned and medium abraded in the grain direction. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.

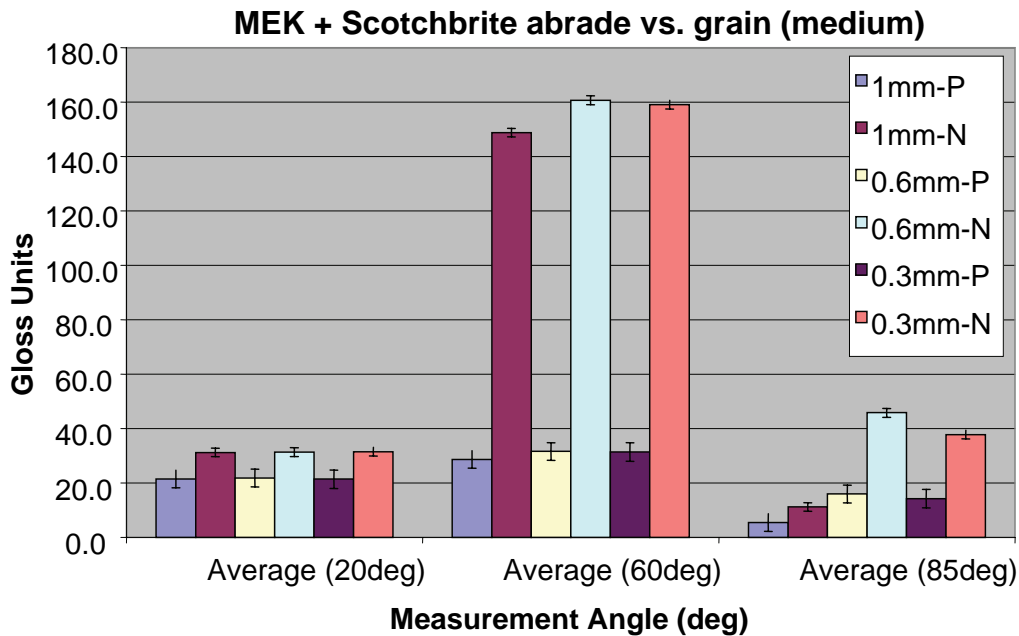


Figure 26 Gloss meter readings for Al2024-T3 clad aluminium that was MEK cleaned and medium abraded against the grain direction. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.

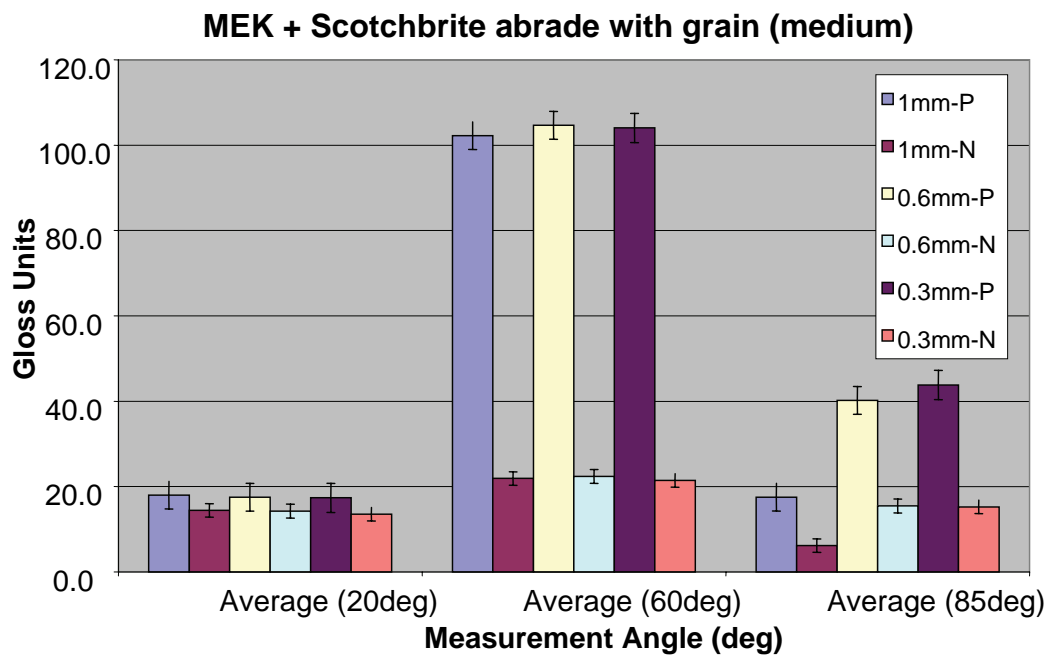


Figure 27 *Gloss meter readings for Al2024-T3 clad aluminium that was MEK cleaned and medium abraded with the grain direction. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.*

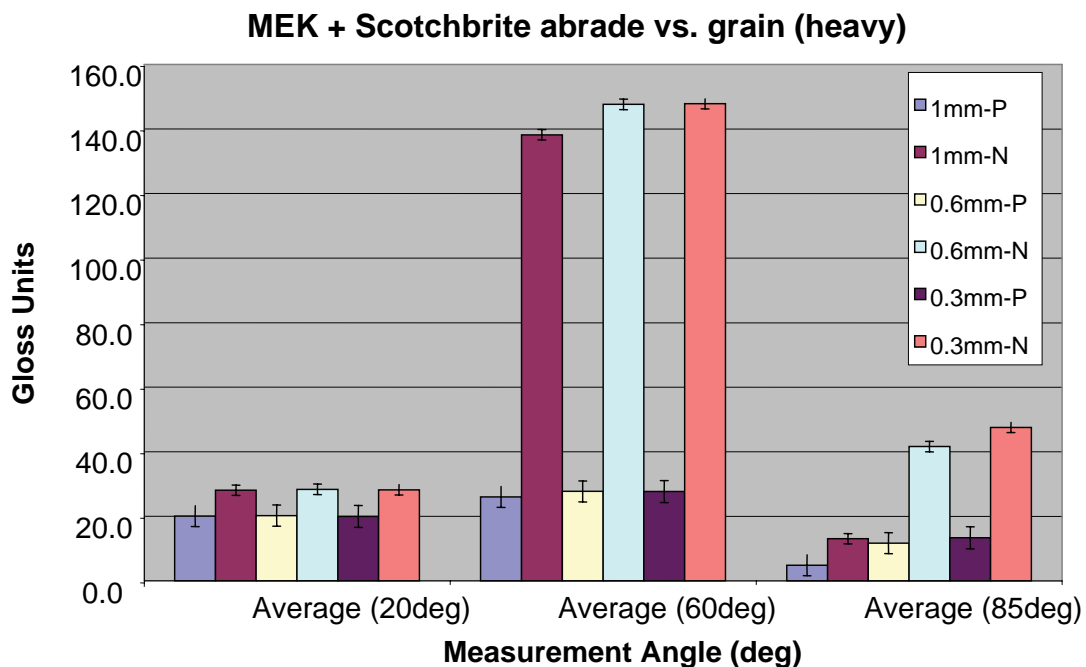


Figure 28 *Gloss meter readings for Al2024-T3 clad aluminium that was MEK cleaned and heavily abraded versus the grain direction. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.*

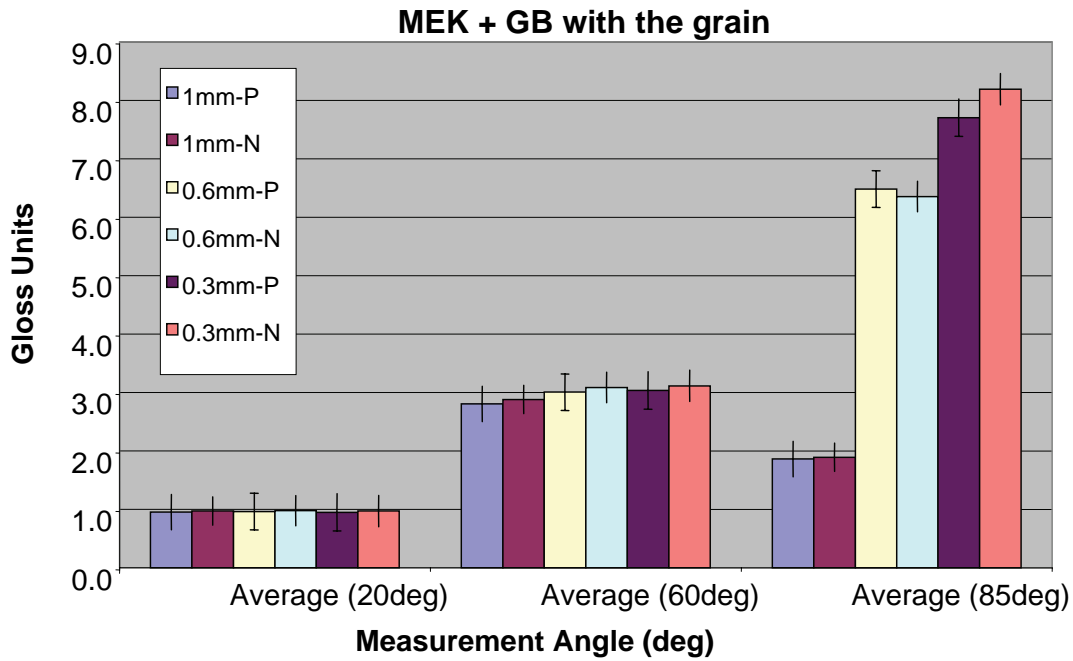


Figure 29 Gloss meter readings for Al2024-T3 clad aluminium that was MEK cleaned and grit-blasted with the grain. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.

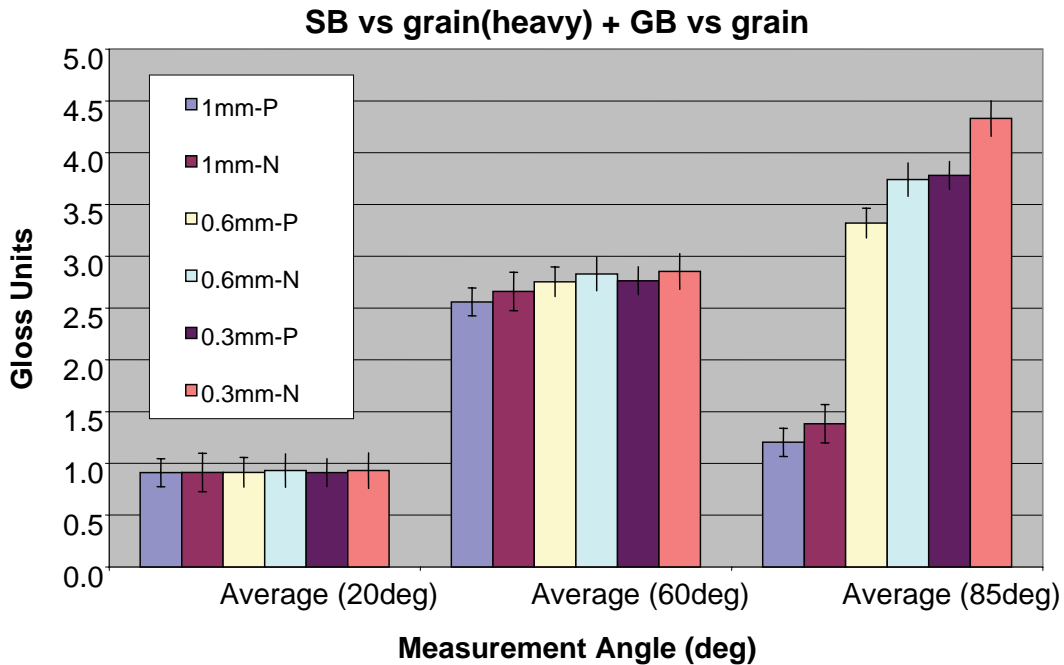


Figure 30 Gloss meter readings for Al2024-T3 clad aluminium that was scotchbrite abraded heavily against the grain and grit-blasted with the grain. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.

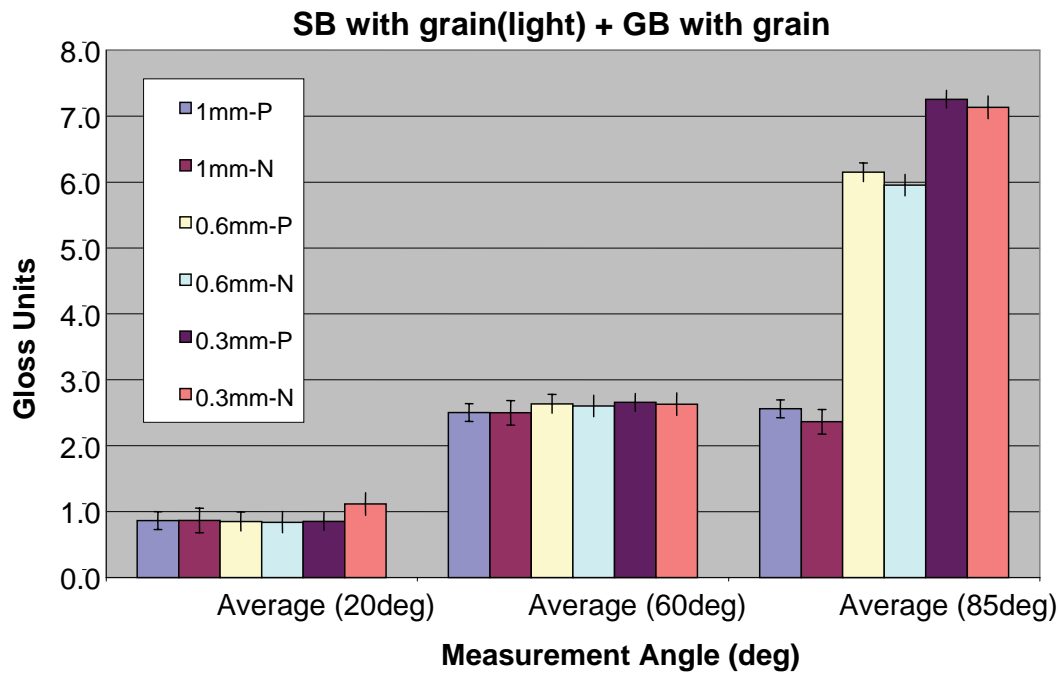


Figure 31 *Gloss meter readings for Al2024-T3 clad aluminium that was scotchbrite abraded heavily against the grain and grit-blasted with the grain. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.*

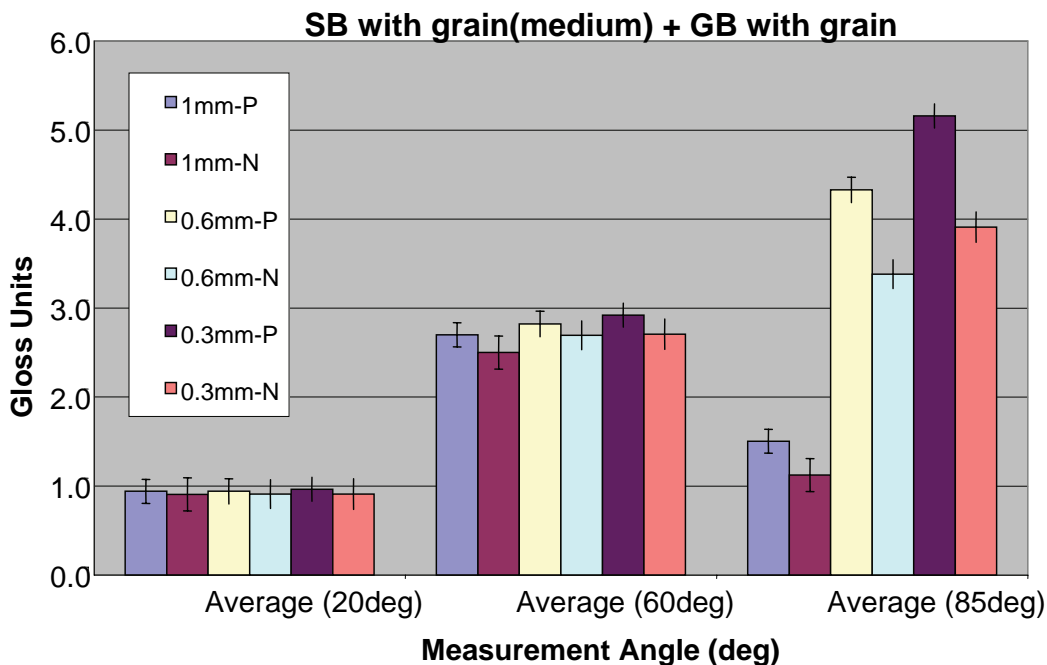


Figure 32 *Gloss meter readings for Al2024-T3 clad aluminium that was scotchbrite abraded with medium pressure with the grain and grit-blasted with the grain. Measurements with the gloss meter were taken at heights of 1, 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.*

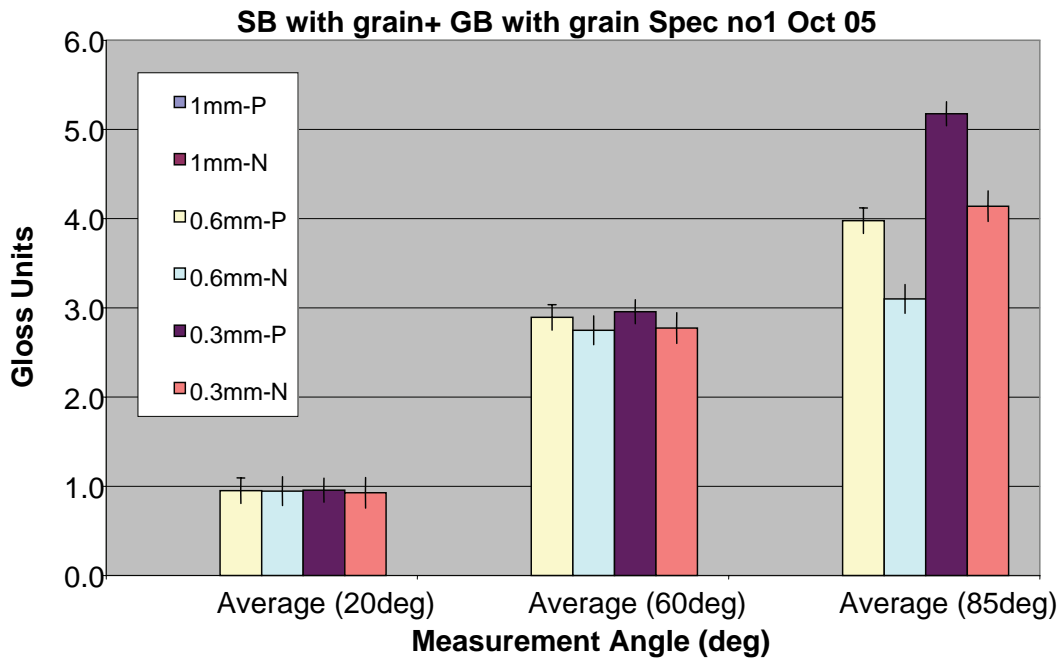


Figure 33 Gloss meter readings for Al2024-T3 clad aluminium that was scotchbrite abraded with the grain and grit-blasted with the grain. Measurements with the gloss meter were taken at heights of 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.

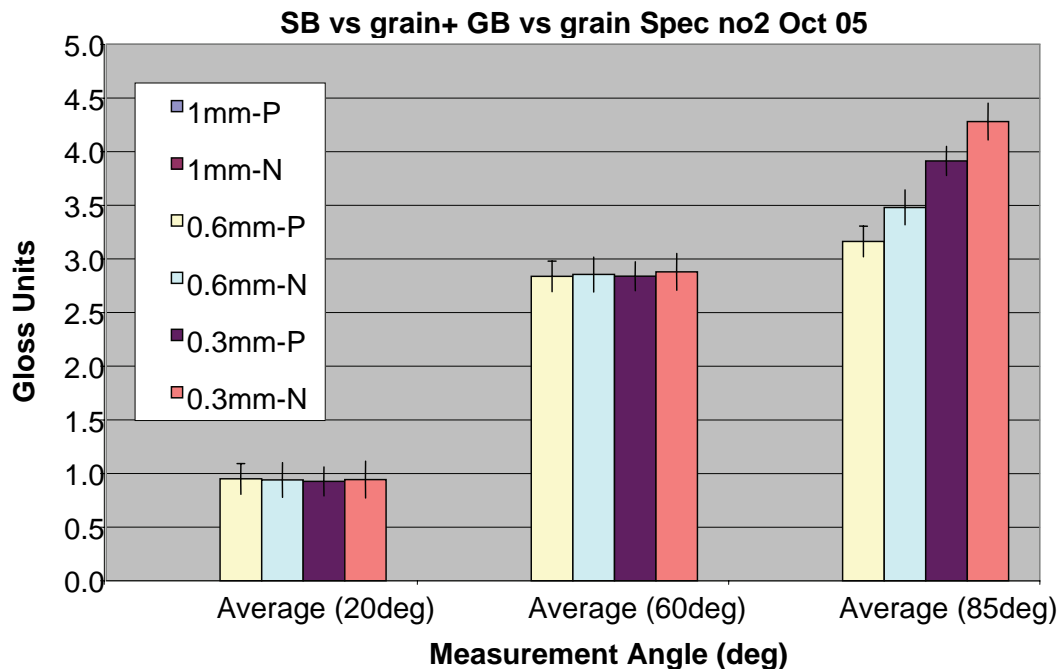


Figure 34 Gloss meter readings for Al2024-T3 clad aluminium that was scotchbrite abraded versus the grain and grit-blasted versus the grain. Measurements with the gloss meter were taken at heights of 0.6 and 0.4mm parallel (P) and normal (N) to the grain direction.

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19. ABSTRACT Adhesively bonded repairs to metallic aircraft structure can be used in a variety of applications to solve difficult maintenance issues where traditional mechanically fastened repairs are often unsatisfactory. For example bonded repairs have been applied to reduce stress intensity in fatigue prone areas of aircraft and, thereby, extend service life of the component, providing substantial maintenance savings. Despite their valuable contribution to aircraft maintenance, bonded repairs are treated as fail-safe items when used on primary aircraft structure. One of the reasons for the lack of credit for bonded repairs is the absence of a reliable non-destructive inspection (NDI) technique that can guarantee bond quality and strength. One solution to reduce this problem is the development of objective prebond inspection techniques that can guarantee the quality and reliability of the critical surface treatment process applied prior to the adhesive bonding operation. The use of a gloss-meter unit and surface quality monitor to quantify the prebond condition of metallic substrates is an effort to further improve the reliability and reproducibility of current bonding operations.					